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FILE 'CAPLUS' ENTERED AT 13:08:00 ON 23 AUG 2004

L1 610 SEA ABB=ON PLU=ON ((DEGRAD? OR DECOMPOS?) (5A) POLYMER?)  
(S) ("TOF SIMS" OR (MASS (2A) SPEC?))

L2 65 L1 AND OLIGOMER?

L3 267 POLYMER? (S) "TOF SIMS"

L4 23 L3 AND DEGRAD?

L2 ANSWER 1 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 2003:656340 CAPLUS

DOCUMENT NUMBER: 139:185627

TITLE: *Method for testing the degradation of a drug in polymeric materials*

INVENTOR(S): *Gardella, Joseph A.; Chen, Jiaying; Hernandez De Gatica, Norma L.;  
Lee, Joo-Woon*

PATENT ASSIGNEE(S): *The Research Foundation of State University of New York,  
USA*

SOURCE: U.S. Pat. Appl. Publ., 25 pp., Cont.-in-part of U.S. Ser. No. 680,701,  
abandoned.

CODEN: USXXCO

PATENT NO. KIND DATE APPLICATION NO. DATE

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US 2003157722 A1 20030821 US 2003-331802 20030407

US 6670190 B2 20031230

PRIORITY APPLN. INFO.: US 1999-157964P P 19991006

US 2000-680701 B2 20001006

AB The present invention provides a novel method for monitoring the surface concentration of a drug in a polymer blend matrix and the reaction kinetics of the biodegradable polymers. Detailed information on surface concentration, degradation rates, degradation kinetics and mechanism, is provided by using Time-of-Flight Secondary Ion Mass Spectrometry (ToF SIMS) measurements. Also provided is a method for determining oligomers in hydrolyzed biodegradable polymers. ToF SIMS results of hydrolyzed polylactide and polyglycolide is provided.

L2 ANSWER 3 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 2002:668117 CAPLUS

DOCUMENT NUMBER: 138:39930

TITLE: "Quantitative TOF-SIMS analysis of oligomeric degradation products at the surface of biodegradable poly(<SYM97>-hydroxy acid)s"

AUTHOR(S): *Lee, Joo-Woon; Gardella, Joseph A.*

CORPORATE SOURCE: Department of Chemistry, State University of New York at Buffalo, Buffalo, NY, USA

**Journal of the American Society for Mass Spectrometry (2002), 13(9), 1108-1119**

CODEN: JAMSEF; ISSN: 1044-0305

PUBLISHER: Elsevier Science Inc.

DOCUMENT TYPE: Journal

LANGUAGE: English

AB This paper reports the development of a new method for quantification of the hydrolytic surface degradation kinetics of biodegradable poly(<SYM97>-hydroxy acids) using time-of-flight secondary ion mass spectrometry (TOF-SIMS). We report results from static SIMS spectra of a series of poly(<SYM97>-hydroxy acids) including poly(glycolic acid), poly(L-lactic acid), and random poly(D,L-lactic acid-co-glycolic acid) hydrolyzed in various buffer systems. The distribution of the most intense peak intensities of ions generated in high mass range of the spectrum reflects the intact degradation products (oligomeric hydrolysis products) of each biodegradable polymer. First, a detailed anal. of the oligomeric ions is given based on rearrangement of the intact hydrolysis products. The pattern of ions can distinguish both degradation-generated intact oligomers and their fragment ion peaks with a variety of combinations of each repeat unit. Then, the integration and summation of the area of all ion peaks with the same number of repeat units is proposed as a measurement that provides a more accurate MW average than the typically used method which counts only the most intense peak. The multiple ion summation method described in this paper would be practical in the improvement of quant. TOF-SIMS studies as a better data reduction method, especially in the surface degradn. kinetics of biodegradable polymers.

REFERENCE COUNT: 36

L2 ANSWER 5 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 2002:641161 CAPLUS

DOCUMENT NUMBER: 137:385183

TITLE: "Thermal and thermoxidative degradation processes in poly(bisphenol a carbonate)"

AUTHOR(S): *Montaudo, Giorgio; Carroccio, Sabrina; Puglisi, Concetto*

CORPORATE SOURCE: Dipartimento di Scienze Chimiche, Universita di Catania, Catania, 95125, Italy

SOURCE: **Journal of Analytical and Applied Pyrolysis (2002), 64(2), 229-247**

CODEN: JAAPDD; ISSN: 0165-2370

PUBLISHER: Elsevier Science B.V.

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Progress in the study of thermal and thermoxidative decomposition processes of poly(bisphenol a carbonate) (PC) was obtained by Matrix assisted laser desorption ionization time of flight (MALDI-TOF) and FAB mass spectroscopic techniques. PC samples were subjected to thermal degradation (under inert atmospheric) heating at 300, 350, 400 and 450 °C. The MALDI mass spectra of the pyrolysis residues obtained at 300 °C showed only a progressive reduction of the abundance of cyclic oligomers and an increase of the molar mass of the PC sample most likely due to post-condensation polymerization reactions. At 350 °C the occurrence of an extensive hydrolysis reaction producing phenol groups was observed. PC chains terminated with phenol groups together with chains bearing Ph and isopropenyl end groups were observed at 400 °C. Condensed aromatic compds. such as xanthenes were also detected in the MALDI spectra of PC samples heated at 400 °C, and they became the most intense species at 450 °C. The FAB-MS spectrum of the pyrolysis residue obtained at 400 °C subjected to

a selective and exhaustive aminolysis reaction showed the presence of compds. containing several consecutive xanthone units. These units undergo crosslinking processes, leading to a graphite-like charred residue (insol.), as the temperature increases. The thermoxidative degradation of PC carried out heating the samples in air at 300 °C for 30, 60, 90, 120, 150 and 180 min produced a reduction of the PC molar mass. The MALDI mass spectra of thermally oxidized PC samples at 300 °C for 90 and 180 min showed the presence of PC chains terminated with methyl-ketone, Ph and phenol groups, most likely formed by the oxidative degradation of the isopropylidene bridge of bisphenol A units. The presence of dehydrogenated structures containing biphenyl linkage may account for the insol. residue produced in these conditions. REFERENCE COUNT: 37

L2 ANSWER 6 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN  
ACCESSION NUMBER: 2002:311711 CAPLUS  
DOCUMENT NUMBER: 137:79343  
TITLE: Thermal decomposition kinetics of functionalized polynorbornene  
AUTHOR(S): Wedlake, Michael D.; Kohl, Paul A.  
CORPORATE SOURCE: School of Chemical Engineering, Georgia Institute of Technology, Atlanta, GA, 30332-0100, USA  
SOURCE: Journal of Materials Research (2002), 17(3), 632-640  
CODEN: JMREEE; ISSN: 0884-2914  
PUBLISHER: Materials Research Society  
DOCUMENT TYPE: Journal  
LANGUAGE: English

AB The mechanism and kinetic parameters for the thermal decomposition of four functionalized addition-polymerized polynorbornenes (PNB) were studied by dynamic and isothermal thermogravimetric analyses and by mass spectrometry. The PNBs are 5-methyl- (MePNB), 5-butyl- (BuPNB), and 5-hexyl-PNB (HxPNB) homopolymers and a copolymer of 5-butyl-norbornene and 5-triethoxysilyl-norbornene (BuTESPNB); the average mol. weight for these polymers is 198,000 (MePNB), 185,000 (BuPNB), 195,000 (HxPNB), and 300,000 (BuTESPNB), as determined by the manufacturer (BF Goodrich Corp.). The dynamic and isothermal thermogravimetric data indicate a first-order degradation reaction mechanism with an activation energy of  $229.6 \pm 12.5$  kJ/mol. Based on the polymer structure, reference mass spectra for related mols., and a cross-comparison of the mass spectra, the backbone, free-radical scission mechanism occurs by cleavage of the linkages between bicyclic rings and the production of volatile monomer and oligomers. The degradation of polynorbornene occurred via a depropagation and transfer reaction process. Initially, the depropagation pathway was preferred, but with increasing conversion, intra- and intermol. hydrogen transfer reactions dominated. The knowledge and control of thermal decomposition of polynorbornenes is of interest in applications such as insulators, optical materials, photoresists, and sacrificial material in fabrication of nanostructures. REFERENCE COUNT: 24

L2 ANSWER 7 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN  
ACCESSION NUMBER: 2001:795249 CAPLUS

DOCUMENT NUMBER: 136:263680

TITLE: Prediction of cleavage sites for polymers in SIMS by MO calculations

AUTHOR(S): Endo, Kazunaka

CORPORATE SOURCE: Dep. Chem., Fac. Sci., Kanazawa Univ., Kanazawa, 920-1192, Japan

SOURCE: Journal of the Mass Spectrometry Society of Japan 2001), 49(4), 135-143

CODEN: JMSJEY; ISSN: 1340-8097

PUBLISHER: Nippon Shitsuryo Bunseki Gakkai

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The cleavage sites of representative polymers in time-of-flight (TOF)- and static(S)-secondary ion mass spectrometry (SIMS) can be estimated from the two-center bond energies of the elec. neutral model oligomers by semiempirical MO calcns. using the AM1 method in MOPAC software. The cleavage of the intramol. bonds may be classified into three cases: (a) the scission can occur in any bonds [polyethylene, poly(vinyl Me ether), polyacrylonitrile, poly(vinyl alc.)]; (b) the cleavage of the main chain occur in any bonds, after pendant groups break first [poly(vinyl chloride), poly(acrylamide)]; (c) the main chain carbons with the pendent group break in any bonds of the main chain [poly(vinyl fluoride), poly(tetrafluoroethylene)]. Examples of the cleavage are also used in secondary pos.-ion fragment spectra of conductive polymers. Furthermore, we analyze the decomposition process of nitrocellulose (NC) due to X-ray induced surface damage by the MO calcns. using the elec. neutral dimer model. The scission of NO<sub>2</sub> groups in the NC is confirmed from the TOF-SIMS measurement.

REFERENCE COUNT: 23

L2 ANSWER 8 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 2001:464149 CAPLUS

DOCUMENT NUMBER: 136:102759

TITLE: Simultaneous TG-MS studies on polymers derived from monomers with a polar group

AUTHOR(S): Chen, Jian Ping; Kodaira, Toshiyuki; Isa, Kimio; Senda, Tetsuya

CORPORATE SOURCE: Department of Materials Science and Engineering, Faculty of Engineering, Fukui University, Fukui, 910-8507, Japan

SOURCE: Journal of the Mass Spectrometry Society of Japan (2001), 49(2), 41-50

CODEN: JMSJEY; ISSN: 1340-8097

PUBLISHER: Nippon Shitsuryo Bunseki Gakkai

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Thermal degradation behavior of polymers derived from monomers with a polar group was investigated by thermogravimetry interfaced to mass spectrometry (TG-MS) to see its applicability to the investigation on thermal degrdns. of polymers. The polymers studied were poly(Me methacrylate) (PMMA), poly(Me acrylate) (PMA), and polyacrylamide (PAM). Decomposition products of PMMA consist exclusively of Me methacrylate monomers that were formed through random scissions of main chains followed by depolymn. Initiation temperature of the random scission of PMMA was strongly influenced by its micro-structures, such as head-to-head and head-to-tail

linkages and vinyl terminated chain ends, which suggests that subtle difference of polymer structures can be elucidated by TG-MS studies. This could be the characteristic feature of TG-MS that differentiates from other anal. method. During thermal degradation of PMA, unbuttoning reactions, some portion of unzipping reactions, and random scission of main and side chains occurred to afford a wide variety of products including monomers, oligomers, and some other unknown substances. Intra- and intermol. imidation reactions between the side chain amide groups proceeded to yield glutarimide rings through main chains and crosslinking, resp., during the initial stage of heat-treatments of PAM. Then, various low mol. weight compds. such as glutarimide and its analogs were formed by the decomposition on further heat-treatment at elevated temperature. The thermal degradation mechanism of these polymers was discussed in terms of the ceiling temperature of the monomers from which these polymers were derived. The results obtained were in good accordance with those reported using other anal. methods, which indicates that TG-MS could be one of the useful techniques for the investigation of thermal degrdns. of polymers.

REFERENCE COUNT: 29

L2 ANSWER 9 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 2001:265713 CAPLUS

DOCUMENT NUMBER: 134:281301

TITLE: *Method for testing degradation of polymeric materials*

INVENTOR(S): *Gardella, Joseph A., Jr.; Chen, Jiaying; Hernandez De Gatica, Norma; Lee, Joo-woon*

PATENT ASSIGNEE(S): *Research Foundation of State University of New York, USA*

SOURCE: *PCT Int. Appl., 41 pp.*

CODEN: PIXXD2

PATENT NO. KIND DATE APPLICATION NO. DATE

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WO 2001025784 A1 20010412 WO 2000-US27735 20001006

PRIORITY APPLN. INFO.: US 1999-157964P P 19991006

AB *The method for determining reaction kinetics of a biodegradable polymer comprises the steps, which are in no particular order, of: selecting a biodegradable polymer; initiating hydrolytic degradation of the biodegradable polymer in vitro; monitoring the biodegradn. process of the biodegradable polymer with a time-of-flight secondary ion mass spectrometry (TOF-SIMS) instrument to measure oligomers generated during the hydrolytic degradation which provides information for kinetics anal. of the biodegradable polymer within a predetd. time frame; calculating the reaction kinetics of the biodegradable polymer. The method can provide detailed information on degradation rates, kinetics and mechanisms of the degradation. REFERENCE COUNT: 11*

L2 ANSWER 10 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 2000:890588 CAPLUS

DOCUMENT NUMBER: 134:237939

TITLE: *Mass spectral analysis of low-temperature pyrolysis products from poly(tetrahydrofuran)*

AUTHOR(S): Lattimer, R. P.  
CORPORATE SOURCE: The BFGoodrich Research and Development Center,  
Brecksville, OH, 44141, USA  
SOURCE: Journal of Analytical and Applied Pyrolysis (2001), 57(1), 57-76  
CODEN: JAAPDD; ISSN: 0165-2370  
PUBLISHER: Elsevier Science B.V.  
DOCUMENT TYPE: Journal  
LANGUAGE: English

AB A poly(tetrahydrofuran) (PTHF) sample of average mol. weight 2000 was subjected to low temperature pyrolysis. The polymer was pyrolyzed in Pyrex tubes (closed at one end) placed at the end of the carrier gas inlet in a gas chromatograph. Pyrolysis was carried out under argon flow in the temperature range 150-350°C. After pyrolysis, the residue in the pyrolysis tube was analyzed by matrix-assisted laser desorption/ionization mass spectrometry (MALDI-MS) and by direct probe chemical ionization (isobutane CI-MS). Eleven series of oligomeric pyrolyzates were characterized by MALDI-MS and CI-MS. Assignment of chemical structures was aided by tandem mass spectrometry (CI-MS/MS) and by deuteration of hydroxyl end groups in the pyrolyzate. Pyrolysis of poly(tetrahydrofuran) ensues about 175°C in inert atmospheric. The initial pyrolysis products all have at least one hydroxyl end group, which is retained from the low mol. weight starting polymer. The other end group is Et ether, Pr ether, Bu ether, or aldehyde. At higher temps. (250-350°C) there is an increasing tendency to form products with a combination of alkyl ether and/or aldehyde end groups. The proportion of products containing the hydroxyl end group diminishes at the higher temps. There is little tendency to form alkenyl ether end groups during PTHF pyrolysis. Only the butenyl ether is observed to any appreciable extent. This is evidently formed mainly via dehydration of oligomers containing a hydroxyl end group. A free radical mechanism is proposed to explain the main degradation products. REFERENCE COUNT: 11

L2 ANSWER 13 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 2000:787588 CAPLUS

DOCUMENT NUMBER: 134:57084

TITLE: "Py-GC/MS an effective technique to characterizing of degradation mechanism of poly(L-lactide) in the different environment"

AUTHOR(S): *Khabbaz, Farideh; Karlsson, Sigbritt; Albertsson, Ann-Christine*

CORPORATE SOURCE: Department of Polymer Technology, Royal Institute of Technology (KTH), Stockholm, S-100 44, Swed.

SOURCE: **Journal of Applied Polymer Science (2000), 78(13), 2369-2378**

CODEN: JAPNAB; ISSN: 0021-8995

PUBLISHER: John Wiley & Sons, Inc.

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The biotic and abiotic degradation of poly (L-lactide) (PLLA) has been studied with pyrolysis gas chromatog. mass spectrometry (Py-GC-MS). A mixed culture of compost micro-organisms was used as the biotic medium. Size-exclusion chromatog. (SEC), gas chromatog.-mass spectrometry (GC-MS), Fourier transform IR spectroscopy (FTIR), and SEM were utilized to monitor the degradation and degradation mechanism. Differences

in pH, mol. weight, surface structure, and degradation mechanisms were noted between sample aged in biotic and abiotic medium. Using fractionated Py-GC-MS at 400 and 500°C, acetaldehyde, acrylic acid, lactoyl acrylic acid, two lactide isomers, and cyclic oligomers up to the pentamer were identified as thermal decomposition products of PLA as well as some other not completely identified products. The ratio of meso-lactide to L-lactide was lower in the sample aged in the biotic media than the abiotic media. This is a result of the preference of the micro-organisms for L-form of lactic acid and lactoyl lactic acid rather than the D-form that in turn influences the formation and the amts. of meso and D,L-lactide during the pyrolysis. Based on SEM micrographs, it was shown that degradation in the biotic medium proceeded mainly via a surface erosion mechanism, whereas bulk erosion was the predominant degradation mechanism in the abiotic medium. The SEC and Py-GC-MS data indicate that degradation was faster in the biotic than in the abiotic sample. REFERENCE COUNT: 25

L2 ANSWER 14 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 2000:526328 CAPLUS

DOCUMENT NUMBER: 133:282173

TITLE: "Mass spectral analysis of low-temperature pyrolysis products from poly(ethylene glycol)"

AUTHOR(S): *Lattimer, R. P.*

CORPORATE SOURCE: The BFGoodrich Research and Development Center, Brecksville, OH, 44141, USA

SOURCE: **Journal of Analytical and Applied Pyrolysis (2000), 56(1), 61-78**

CODEN: JAAPDD; ISSN: 0165-2370

PUBLISHER: Elsevier Science B.V.

DOCUMENT TYPE: Journal

LANGUAGE: English

AB A poly(ethylene glycol) sample of average mol. weight 2000 was subjected to low temperature pyrolysis. The polymer was pyrolyzed in Pyrex tubes (sealed at one end) placed at the end of the carrier gas inlet in a gas chromatograph. Pyrolysis was carried out under Ar flow at 150-325°. After pyrolysis, the residue was analyzed by MALDI mass spectrometry (MALDI-MS) and by direct probe chemical ionization-MS (CI-MS). Assignment of chemical structures was aided by tandem mass spectrometry (CI-MS/MS) and by deuteration of the terminal OH groups in the pyrolyzate. At the lowest temps. the dominant oligomeric products have OH and OEt end groups. At higher temps., OMe and vinyl ether end groups become more abundant. The pyrolysis expts. indicate that the decomposition scheme is free radical in nature. Cleavage of CO bonds is preferred at the onset of pyrolysis; H-abstraction following CO homolysis produces OH and OEt end groups. At higher temps., the abundance of OMe and OEt end groups becomes more balanced as relatively more C-C cleavage occurs. The increasing abundance of vinyl ether end groups at higher pyrolysis temps. is primarily due to dehydration of hydroxyl end groups. REFERENCE COUNT: 9

L2 ANSWER 15 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 2000:134104 CAPLUS

DOCUMENT NUMBER: 132:237730

TITLE: Analysis of volatile radiolysis products in gamma-irradiated LDPE and polypropylene films by thermal desorption-gas chromatography-mass spectrometry  
AUTHOR(S): Buchalla, Rainer; Boess, Christian; Bogl, Klaus Werner  
CORPORATE SOURCE: Federal Institute for Health Protection of Consumers and Veterinary Medicine (Bg VV), Berlin, D-14195, Germany  
SOURCE: Applied Radiation and Isotopes (2000), 52(2), 251-269  
CODEN: ARISEF; ISSN: 0969-8043

PUBLISHER: Elsevier Science Ltd.

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Low-mol.-weight ("volatile") radiolysis products of low-d. polyethylene (LDPE) and polypropylene (PP) films were investigated by thermal desorption-(TDS)-GC-MS after absorbed doses of up to 25 kGy. The films produce fingerprint chromatograms with highly characteristic patterns of groups of radiation-induced peaks; these are mainly hydrocarbons, aldehydes, ketones, and carboxylic acids with concns. (after 25 kGy) ca one order of magnitude below that of the residual hydrocarbons (oligomers). PP addnl. produces very substantial amts. of three degradation products of phenol-type antioxidants. The low mol.-weight (MW) radiolysis products are retained for considerable times in LDPE films and they are retained in PP films much longer than had been expected. Besides product identification, the following topics are addressed: Effects of the absorbed dose and the desorption temperature; comparison of several com. (proprietary) films; high-temperature thermal desorption; the question whether TDS analyzes radiation-induced artifacts rather than genuine products; the possible existence of cyclic radiolysis products; the possibility of identifying an LDPE film as irradiated after a dose of only 1 kGy; and atypical trace fragments of antioxidants. Finally, the geometry and efficiency of the thermal desorption system is briefly discussed, and the implications of our findings for irradiation detection and for the safety of irradiated materials are considered. REFERENCE COUNT: 61

L2 ANSWER 16 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1999:780493 CAPLUS

DOCUMENT NUMBER: 132:3711

TITLE: Mass spectrometric analysis of an epoxyphenolic adhesive in the bulk as well as in contact with a metal surface

AUTHOR(S): Zaitsev, A. L.; Pleskachevsky, Yu. M.

CORPORATE SOURCE: V. A. Belyi Metal Polymer Research Institute, Belarus Natl. Acad. Sci., Gomel, 246652, Belarus

SOURCE: Journal of Adhesion Science and Technology (1999), 13(11), 1295-1306

CODEN: JATEE8; ISSN: 0169-4243

PUBLISHER: VSP BV

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The chemical composition of low-mol.-weight products of polycondensation during the curing of an epoxy-phenolic adhesive both in the bulk and in a thin layer in contact with a metal was studied using pyrolytic mass spectrometry. It was found that the volatile products from the adhesive bulk differed greatly from those released from the



thin oligomer layer bordering the metal surface. The difference in chemical composition of the polycondensation products near the metal surface compared with those in the bulk is related to simultaneously occurring thermal desorption of gases from the metal surface. Due to the dissoln. of desorption products in the uncured adhesive surface layer, a change is observed in the process of direction of polycondensation and in the chemical composition of the thin subsurface adhesive layer. The greatest effect on the adhesive surface layer structure and properties is exerted by the carbon mono- and dioxides desorbed from the metal. The metal surface serves as a catalyst for carboxylation and carbonylation taking place near the interface. Thermodesorption of phys. and chemical adsorbed mols. and the competing process of solvent absorption by metal structural defects are the possible causes for the onset of microdefects in the epoxy-phenolic adhesive layers contacting the metal. REFERENCE COUNT: 14

L2 ANSWER 17 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1999:685649 CAPLUS

DOCUMENT NUMBER: 132:251525

TITLE: "Repetitive injection gas chromatography/mass spectrometry for evolved gas analysis"

AUTHOR(S): *White, Robert L.; Negelein, Darrel L.; Bonnet, Edouard; Lin, Rong*

CORPORATE SOURCE: Department of Chemistry and Biochemistry, University of Oklahoma, Norman, OK, 73019, USA

SOURCE: **Current Topics in Analytical Chemistry (1998), 1, 93-105**

CODEN: CACHFQ

PUBLISHER: Research Trends

DOCUMENT TYPE: Journal

LANGUAGE: English

AB An interface and gas chromatograph oven are described that permit repetitive separation and mass spectrometric anal. of evolved gases generated during solid sample thermal decompns. Examples of the use of this apparatus for identifying the volatile products generated by heating poly(vinyl butyral), thermally desorbing 1-butene oligomers from sulfated zirconia, and by cracking poly(styrene) with HY zeolite are described. Evolved gas analyses employing rapid temperature programmed capillary gas chromatog. and subambient column temps. are demonstrated. Species-specific evolution profiles are generated by integrating peaks that correspond to the same substance in successive chromatograms. REFERENCE COUNT: 77

L2 ANSWER 18 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1999:646446 CAPLUS

DOCUMENT NUMBER: 131:352011

TITLE: "Time-of-Flight Secondary Ion Mass Spectrometry Studies of in Vitro Hydrolytic Degradation of Biodegradable Polymers"

AUTHOR(S): *Chen, Jiaxing; Gardella, Joseph A., Jr.*

CORPORATE SOURCE: Department of Chemistry, SUNY at Buffalo, Buffalo, NY, 14260-3000, USA

SOURCE: **Macromolecules (1999), 32(22), 7380-7388**

CODEN: MAMOBX; ISSN: 0024-9297

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The in vitro hydrolytic degradation at the surface of six biodegradable polymers, namely polyglycolic acid, poly(lactic acid), random poly(lactic-co-glycolic) acid, poly(sebacic acid), and two random copolymer poly(fumaric-co-sebacic) acid of varying composition was studied using time-of-flight secondary ion mass spectrometry (TOF-SIMS). A distribution of hydrolysis products, namely oligomer mols., were observed as a series of intact mol. ions. In most cases, the mol. ion peak in each repeating pattern is the most intense peak. Anal. of the intensities of the mol. ions in the distribution allows chemical kinetic information to be obtained. These data can be used for rapid screening of formulations and preparation of new materials. REFERENCE COUNT: 60

L2 ANSWER 19 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1999:173833 CAPLUS

DOCUMENT NUMBER: 130:325470

TITLE: "Thermal Decomposition Processes in Aromatic Polycarbonates Investigated by Mass Spectrometry"

AUTHOR(S): *Puglisi, Concetto; Sturiale, Luisa; Montaudo, Giorgio*

CORPORATE SOURCE: Istituto per la Chimica e la Tecnologia dei Materiali Polimerici, Consiglio Nazionale delle Ricerche, Catania, 95125, Italy

SOURCE: **Macromolecules (1999), 32(7), 2194-2203**

CODEN: MAMOBX; ISSN: 0024-9297

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The thermal decomposition pathways leading to the formation of volatile compds. and to char residue in poly(bisphenol A carbonate) (PC), poly(resorcinol carbonate) (PRC), and poly(hydroquinone carbonate) (PHC) were studied by mass spectrometry. The structure of the volatile compds. obtained at 300-700° by direct pyrolysis mass spectrometry (DPMS), suggests that thermal decomposition occurs by different pyrolysis processes. In the initial stage, cyclic oligomers are formed by an intramol. exchange reaction, whereas evolution of CO<sub>2</sub> and H<sub>2</sub>O takes place over the pyrolysis temperature range and leads to formation of ether bridges (decarboxylation) and phenolic end groups (hydrolysis). A disproportionation reaction of the BPA isopropylidene bridges of PC itself takes place at higher temperature yielding Ph and isopropylidene end groups, whereas pyrolysis products containing dibenzofuran units are formed by dehydrogenation of ether units. The formation of compds. containing xanthone and fluorenone units, most likely generated by isomerization of the aromatic carbonate functional groups and successive condensation reaction, was observed. Isothermal pyrolysis of PC was carried out at 350 and 400°, followed by exhaustive and selective aminolysis of the residual carbonate functional groups in the pyrolysis residue. The aminolyzed residue was then analyzed by fast atom bombardment (FAB) mass spectrometry to detect the compds. eventually formed by mol. rearrangements of PC chains. The FAB-MS spectra showed the presence of compds. containing several consecutive xanthone and ether units, indicating that at this temperature the

isomerization and the condensation processes leading to these structures are quite extensive. These units undergo aromatization and crosslinking processes, leading to a graphite-like charred residue as the temperature increases . REFERENCE COUNT: 30

L2 ANSWER 21 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN  
ACCESSION NUMBER: 1997:76081 CAPLUS  
DOCUMENT NUMBER: 126:145251  
TITLE: "Pyrolysis field ionization mass spectrometry of hydrocarbon polymers"  
AUTHOR(S): *Lattimer, Robert P.*  
CORPORATE SOURCE: The BFGoodrich Research and Development Center,  
Brecksville, Ohio 44141, USA  
SOURCE: **Journal of Analytical and Applied Pyrolysis (1997), 39(2), 115-127**  
CODEN: JAAPDD; ISSN: 0165-2370  
PUBLISHER: Elsevier  
DOCUMENT TYPE: Journal  
LANGUAGE: English

AB Two diene rubbers, polybutadiene (PB) and polyisoprene (PI), were studied using pyrolysis-field ionization-mass spectrometry (Py-FIMS). These diene rubbers yielded only one series of intense oligomers at lower masses (up to .apprx.800 Da); the pyrolyzates are multiples of the monomer unit. The higher-mass pyrolysis products are only observable during the initial stage of decomposition (.apprx.310-325°). The major features of the pyrolysis spectra for the two polymers can be interpreted in terms of a free radical degradation mechanisms. The production of long sequences of pyrolyzate oligomers may be explained in terms of main-chain scission followed by intramol. radical transfer (backbiting) reactions and <SYM98>-scission to produce volatile pyrolyzates. Polynorbornene (PN) was also studied as an example of a polymer with a cyclic hydrocarbon monomer unit. The Py-FI mass spectrum is rather complex, but the most prominent pyrolyzates are norbornene oligomers. As with PB and PI, the thermal decomposition of PN can be explained by a free radical degradation chemical

L2 ANSWER 22 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN  
ACCESSION NUMBER: 1996:531652 CAPLUS  
DOCUMENT NUMBER: 125:196603  
TITLE: "Thermal degradation processes in poly(xylylene sulfides) investigated by comparative direct pyrolysis MS and flash pyrolysis GC/MS experiments"  
AUTHOR(S): *Montaudo, Giorgio; Puglisi, Concetto; de Leeuw, Jan W.; Hartgers, Walter; Kishore, K.; Ganesh, K.*  
CORPORATE SOURCE: Dipartimento di Scienze Chimiche, Universita di  
Catania, Catania, 6-95125, Italy  
SOURCE: **Macromolecules (1996), 29(20), 6466-6474**  
CODEN: MAMOBX; ISSN: 0024-9297  
PUBLISHER: American Chemical Society  
DOCUMENT TYPE: JournalLANGUAGE: English  
AB The thermal degradation processes of two sulfur polymers, poly(xylylene sulfide) (PXM) and poly(xylylene disulfide) (PXD), were investigated in parallel by direct pyrolysis mass spectrometry (DPMS) and flash pyrolysis GC/MS (Py-GC/MS).

Thermogravimetric data showed that these polymers decompose with two sep. steps in the temperature ranges of 250-280 and 600-650, leaving a high amount of residue (apprx. 50% at 800°). The pyrolysis products detected by DPMS in the first degradation step of PXM and PXD were terminated by three types of end groups, -CH<sub>3</sub>, -CH<sub>2</sub>SH, and -CH<sub>2</sub>S, originating from thermal cleavage reactions involving a series of homolytic chain scissions followed by hydrogen transfer reactions, generating several oligomers containing some intact xylylene sulfide repeating units. The presence of pyrolysis compds. containing some stilbene-like units in the first degradation step has also been observed. Their formation has been accounted for with a parallel cleavage involving the elimination of H<sub>2</sub>S from the PXM main chains. These unsatd. units can undergo crosslinking at higher temps., producing the high amount of char residue observed. The thermal degradation compds. detected by DPMS in the second decomposition step at apprx. 600-650° were constituted of condensed aromatic mols. containing dihydrophenanthrene and phenanthrene units. These compds. might be generated from the polymer chains containing stilbene units, by isomerization and dehydrogenation reactions. The pyrolysis products obtained in the Py-GC/MS of PXM and PXD at 610° are almost identical. The relative abundance in the pyrolyzate and the spectral properties of the main pyrolysis products were found to be in generally good agreement with those obtained by DPMS. Polycyclic aromatic hydrocarbons (PAHs) were also detected by Py-GC/MS but in minor amts. with respect to DPMS. This apparent discrepancy was due to the simultaneous detection of PAHs together with all pyrolysis products in the Py-GC/MS, whereas in DPMS they were detected in the second thermal degradation step without the greatest part of pyrolysis compds. generated in the first degradation step. The results obtained by DPMS and Py-GC/MS expts. showed complementary data for the degradation of PXM and PXD and, therefore, allowed the unequivocal formulation of the thermal degradation mechanism for these sulfur-containing polymers.

L2 ANSWER 23 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1996:222078 CAPLUS

TITLE: "Polymer characterization by electrospray ionization mass spectrometric techniques"

AUTHOR(S): *Kemp, T. J.; Barton, Zachary; Mahon, A.; Buzy, A.; Jennings, K. R.*

CORPORATE Dept. Chemistry, University Warwick, Coventry, CV4 7AL, UK

SOURCE: **Book of Abstracts, 211th ACS National Meeting, New Orleans, LA, March 24-28 (1996), POLY-343. American Chemical Society: Washington, D. C.**

CODEN: 62PIAJ

DOCUMENT TYPE: Conference; Meeting Abstract

LANGUAGE: English

AB Electrospray Ionization Mass Spectrometry (ESI-MS) has been applied to the following structural and reaction characteristics of linear polymers: 1) thermal degradation (of polyethers and polysulfides) 2) the presence of cyclic oligomers (in poly(3-nitratomethyl-3-methyloxetane)) 3) the presence of mers containing addnl. oxyalkylene groups (in polysulfides) 4) the gas-phase complexation of cyclic oligomers with alkali metal cations of differing size 5) identification of end-groups (of polysulfides and in poly(3-nitratomethyl-3-methyloxetane)). Assignments of structures are confirmed in many cases by collision-induced decomposition and precursor-ion expts.

L2 ANSWER 24 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN  
ACCESSION NUMBER: 1996:64469 CAPLUS  
DOCUMENT NUMBER: 124:88097  
TITLE: Direct pyrolysis mass spectrometry study of thermal decomposition of poly(<SYM98>-hydroxyalkanates)  
AUTHOR(S): Xie, Hongwei; Liu, Shuying; Yin, Jinghua; Mo, Zhishen  
CORPORATE SOURCE: Changchun Inst. Applied Chem., Academia Sinica, Changchun, 130022, Peop. Rep. China  
SOURCE: Gaofenzi Xuebao (1995), (6), 648-52  
CODEN: GAXUE9; ISSN: 1000-3304  
PUBLISHER: Kexue  
DOCUMENT TYPE: Journal  
LANGUAGE: Chinese

AB Thermal decomposition process in poly(<SYM98>-hydroxybutyrate) (PHB) and poly(<SYM98>-hydroxybutyrate-co-<SYM98>-hydroxyvalerates) [P(HB-co-HV)s] with composition ratios HB/HV of 96:4 and 82:18 was investigated by direct pyrolysis mass spectrometry (DPMS). It can provide up to heptamer fragments and estimate the composition and sequence distribution of copolymers. The thermal degradation of PHB and P(HB-co-HV) occurs quite selectively through a <SYM98>-hydrogen transfer process, with formation of oligomers bearing carboxyl and olefin end-groups. Oligomeric products corresponding to dehydrated monomer up to heptamer were also identified both in the case of PHB and P(HB-co-HV).

L2 ANSWER 25 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN  
ACCESSION NUMBER: 1996:49544 CAPLUS  
DOCUMENT NUMBER: 124:88084  
TITLE: "Plasmaoxidative and chemical degradation of poly(ethylene terephthalate) studied by matrix-assisted laser desorption/ionization mass spectrometry"  
AUTHOR(S): Weidner, St.; Kuehn, G.; Friedrich, J.; Schroeder, H.  
CORPORATE SOURCE: Federal Inst. Mater. Res and Testing (BAM), Berlin, 12205, Germany  
SOURCE: **Rapid Communications in Mass Spectrometry** (1996), 10(1), 40-6  
CODEN: RCMSEF; ISSN: 0951-4198  
PUBLISHER: Wiley  
DOCUMENT TYPE: Journal  
LANGUAGE: English

AB A model system of linear ethylene glycol-terminated poly(ethylene terephthalate) oligomers of the general formula H-[GT]<sub>n</sub>-G (where G is an ethylene glycol unit and T represents a terephthalic acid unit) was synthesized and exposed to an oxygen plasma. The degradation of the oligomers was investigated by matrix-assisted laser desorption/ionization mass spectrometry (MALDI-MS). The results were compared to a chemical degradation of com. poly(ethylene terephthalate) which had been exposed to an aqueous solution of natural chalk for many years. In both cases an ester scission process was found which generates terephthalic acid-terminated oligomers (H-[GT]<sub>m</sub>-OH) and decomp. linear ethylene glycol-terminated oligomers (H-[GT]<sub>m</sub>-G). The scission of the

ester bonds during the chemical treatment addnl. leads to the formation of T-[GT]<sub>m</sub>-OH-like oligomers and to a strong decrease of the number of cyclic oligomers ([GT]<sub>m</sub>). Furthermore during the plasma treatment an addnl. formation of [GT]<sub>m</sub>-G like cyclic oligomers was observed

L2 ANSWER 26 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1995:984851 CAPLUS

DOCUMENT NUMBER: 124:88061

TITLE: "Thermal degradation of polymers in the melt. 1. Characterization of volatile oligomers formed by thermal degradation of polyisobutylene"

AUTHOR(S): *Sawaguchi, Takashi; Takesue, Tomoyuki; Ikemura, Tadashi; Seno, Manabu*

CORPORATE SOURCE: Dep. Ind. Chem., Coll. Sci. Technol., Nihon Univ., Tokyo, 101, Japan

SOURCE: **Macromolecular Chemistry and Physics** (1995), 196(12), 4139-57

CODEN: MCHPES; ISSN: 1022-1352

PUBLISHER: Huethig & Wepf

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The chemical structures of a number of components of volatile oligomers including (n + 2)-mers (n <SYM179> 0) produced by thermal degradation of Vistanex L polyisobutylene were systematically determined by high-resolution capillary gas chromatog./mass spectrometry (GC/MS). The total ion current (TIC) chromatogram consists of about 100 peaks ranging from dimers (2-mers, n = 0) to dodecamers (12-mers, n = 10) and most of the main peaks are classified into four types of terminal monoolefins: a trisubstituted olefin with a tert-Bu end, a vinylidene olefin with a tert-Bu end, a trisubstituted olefin with an iso-Pr end, and a vinylidene olefin with an iso-Pr end. The formation of these monoolefins is reasonably interpreted by intramol. hydrogen abstractions (back-biting) of primary and tertiary terminal macroradicals and subsequent <SYM98> scissions at the inner position of the main chain. In all the chromatograms of each (n + 2)-mer (n <SYM179> 1), the retention times of terminal trisubstituted types of monoolefins were shorter than those of terminal vinylidene types of monoolefins, in contrast to the elution order of dimers. The relative intensities between the interesting peaks of each (n + 2)-mer (n <SYM179> 1) clearly represent that back-biting more predominantly occurs at the methylene hydrogen rather than at the Me group consistent with the difference in the bond dissociation energy of the H-C bond of interest, as opposed to the steric hindrance mechanism.

L2 ANSWER 27 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1995:716699 CAPLUS

DOCUMENT NUMBER: 123:84244

TITLE: "Photocatalytic Degradation of Secondary Alcohol Ethoxylate: Spectroscopic, Chromatographic, and Mass Spectrometric Studies"

AUTHOR(S): *Sherrard, Kim B.; Marriott, Philip J.; Amiet, R. Gary; Colton, Ray; McCormick, Malcolm J.; Smith, Geoff C.*

CORPORATE SOURCE: Department of Applied Chemistry, RMIT, Melbourne, 3001, Australia

SOURCE: **Environmental Science and Technology** (1995), 29(9), 2235-42

CODEN: ESTHAG; ISSN: 0013-936X

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Secondary alc. ethoxylate (SAE) was photocatalytically degraded using a suspension of TiO<sub>2</sub> particles irradiated with long wavelength UV light. High starting concns. of SAE were used (ca. 2000 mg/L), and changes in both SAE oligomer distribution and production of intermediates were monitored by using GC, GC/MS, NMR, and ESMS. Initial preferential cleavage of the ethoxyl groups over HO<SYM183> reactions with the aliphatic portions of the mol. is followed by cleavage at the secondary carbon. Relatively hydrophilic intermediates remained in solution, while the hydrophobic products adhered to the TiO<sub>2</sub>, causing agglomeration of the particles.

L2 ANSWER 28 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1995:554624 CAPLUS

DOCUMENT NUMBER: 122:315554

TITLE: Pyrolysis field ionization mass spectrometry of polyolefins

AUTHOR(S): Lattimer, Robert P.

CORPORATE SOURCE: The BF Goodrich Research and Development Center, 9921 Brecksville Road, Brecksville, OH, 44141, USA

SOURCE: **Journal of Analytical and Applied Pyrolysis** (1995), 31, 203-25

CODEN: JAAPDD; ISSN: 0165-2370

PUBLISHER: Elsevier

DOCUMENT TYPE: Journal

LANGUAGE: English

AB This report describes the pyrolysis field ionization mass spectrometric (Py-FI-MS) anal. of five polyolefins: polyethylene, polypropylene, poly(1-butene), poly(4-methyl-1-pentene), and polyisobutylene. Volatile pyrolyzates were typically observed at most or all carbon nos. to masses well above 1000 Daltons (Da). The distributions of pyrolyzates showed some similarities between the various polymers. Free radical degradation mechanisms have been proposed to explain the patterns of volatile pyrolyzates. These mechanisms are characterized by three principal reaction types: (a) main-chain cleavage to form chain-terminus radicals; (b) intramol. radical transfer (backbiting) to form internal radicals; (c) <SYM98>-scission to form both volatile products and terminally unsatd. polymer residues. All five polymers yielded only one major series of oligomers at lower masses (up to 600 Da or so); these are 1-alkene pyrolyzates. At higher masses (up to 1600 Da or so), most of the polymers yielded intense dialkene volatile pyrolyzates. These results suggest that other hydrocarbon polymers, including copolymers, might also be analyzed successfully by Py-FI-MS.

L2 ANSWER 29 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1995:437524 CAPLUS

DOCUMENT NUMBER: 122:188963

TITLE: Direct pyrolysis mass spectrometric study of thermal decomposition of some electrically conducting polymers

AUTHOR(S): Xie, Hong-Wei; Liu, Shu-Yin; Wang, Li-Xiang  
CORPORATE SOURCE: Changchun Inst. Applied Chem., Chinese Academy Sci.,  
Changchun, 130022, Peop. Rep. China  
SOURCE: Huaxue Xuebao (1995), 53(2), 173-7  
CODEN: HHHPA4; ISSN: 0567-7351  
PUBLISHER: Kexue  
DOCUMENT TYPE: Journal  
LANGUAGE: Chinese  
AB Thermal decomposition processes of poly(thio-1,4-phenylene) (PPS),  
polythiophene, (PT) and polyaniline (PAn) were investigated by direct pyrolysis mass  
spectrometry. PPS pyrolyzed into linear and cyclic oligomers but PT and PAn only  
pyrolyzed into linear oligomers.

L2 ANSWER 30 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN  
ACCESSION NUMBER: 1994:681407 CAPLUS  
DOCUMENT NUMBER: 121:281407  
TITLE: Pyrolysis-gas chromatography-mass spectrometry of poly(dialkylsilylenes)  
AUTHOR(S): Blazso, Marianne  
CORPORATE SOURCE: Research Laboratory for Inorganic Chemistry, Hungarian  
Academy of Sciences, Budaoersi ut 45, Budapest, H-1112, Hung.  
SOURCE: Journal of Chromatography, A (1994), 683(1), 115-24  
CODEN: JCRAEY; ISSN: 0021-9673  
PUBLISHER: Elsevier  
DOCUMENT TYPE: Journal  
LANGUAGE: English  
AB A series of silylene copolymers with di-n-alkyl substituent groups varying from di-  
Me to di-n-hexyl were pyrolyzed in an anal. pyrolyzer coupled to a capillary GC-MS  
system. The thermal decomposition of the silylene copolymers begins at about 200° and  
proceeds via cyclic oligomer formation. The pyrolysis at 300° of those copolymers  
which contain dimethylsilylene units lead to tetracycles and pentacycles, but those which  
have Et or longer alkyl substituents decompose exclusively to tetracycles. The  
copolymer composition and structure were evaluated from the pyrolysis product  
distribution data. Mass spectra and GC retention indexes were obtained on several di-n-  
alkylsilylene cyclotetramers and cyclopentamers.

L2 ANSWER 31 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN  
ACCESSION NUMBER: 1994:165176 CAPLUS  
DOCUMENT NUMBER: 120:165176  
TITLE: "Thermal degradation of poly(phenylene sulfide) as monitored by pyrolysis-  
GC/MS"  
AUTHOR(S): *Budgell, D. R.; Day, M.; Cooney, J. D.*  
CORPORATE SOURCE: Inst. Environ. Chem., Natl. Res. Counc. Canada, Ottawa,  
ON, K1A 0R6, Can.  
SOURCE: **Polymer Degradation and Stability (1994), 43(1), 109-15**  
CODEN: PDSTDW; ISSN: 0141-3910  
DOCUMENT TYPE: Journal



LANGUAGE: English

AB The thermal degradation of two com. samples of poly(phenylene sulfide) was studied over a range of pyrolytic temps. using flash pyrolysis-gas chromatog./mass spectrometry. At the lower pyrolysis temps. (up to about 550°) the major volatile pyrolysis products detected were the cyclic tetramer and the linear trimers and dimers, formed as a result of random scission followed by depolymn. and cyclization. Under more severe pyrolysis conditions (550-800°) the concns. of the oligomers start to decrease and those of benzene and benzenethiol increase. In addition, at these intermediate temps. of 550-650° it appears that rearrangement reactions are occurring followed by cyclization to give rise to benzothiophenes and some naphthothiophenes. The predominant degradation process, however, appears to be random chain scission followed by cyclization at the lower temps. and depolymn. at the higher temps.

L2 ANSWER 32 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1993:428776 CAPLUS

DOCUMENT NUMBER: 119:28776

TITLE: "Linear-temperature programmed pyrolysis of thermoresistant polymers - mass and FTIR spectrometries. 2. Aromatic polyesters and copolyesters"

AUTHOR: *Hummel, Dieter O.; Neuhoff, Ulrich; Bretz, Anke; Duessel, Heinz Juergen*

CORPORATE SOURCE: Inst. Phys. Chem., Univ. Koeln, Cologne, D-5000/41, Germany

SOURCE: **Makromolekulare Chemie (1993), 194(6), 1545-59**

CODEN: MACEAK; ISSN: 0025-116X

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Linear-temperature controlled pyrolysis with subsequent anal. of the pyrolyzates (volatiles and residue) by 18V electron-impact mass spectrometry and FTIR spectrometry, resp., yields optimal informations on the thermal degradation mechanisms of heat-resistant polymers. Poly(4-oxybenzoyl) first (675 K) releases residual acetoxybenzoic acid, solvent (hexahydroterphenyl), phenol, and oligomers. In the temperature range of maximum decomposition ( $T_{\max} = 815$  K) several consecutive processes take place. At 760 K, monomer (oxybenzoyl) and 2 series of oligomers with different endgroups are set free. At 815 K, formation of phenol, and at 845 K, formation of di-Ph ether are the dominating processes. The maximum of CO<sub>2</sub> and CO release are at 790 K and 825 K, resp. In the residue, diaryl ketone, 9-fluorenone, and phenolic structures are observed. Mechanisms including Fries-analog rearrangements are discussed. A typical poly(4-oxybenzoyl-ran-2-oxy-6-naphthoyl) ("Vectra A 950") with 73 mol.% oxybenzoyl units produces, during the evaporation phase (640 K) and in addition to oxybenzoyl monomer and oligomers, oxynaphthoyl and a series of hetero-fragments containing 1 oxynaphthoyl unit and 1 or 2 oxybenzoyl units. At 785 K (close to  $T_{\max}$ ), the formation of phenol, CO<sub>2</sub>, CO and oxybenzoyl is predominant. In the residue, the formation of diaryl ketone, dibenzofuran, and 9-fluorenone structures is observed. Mechanisms are discussed in analogy to the degradation of poly(4-oxybenzoyl).

L2 ANSWER 33 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1993:409431 CAPLUS

DOCUMENT NUMBER: 119:9431

TITLE: Structural characterization of poly(N-alkyl-4-vinylpyridinium) triflates using pyrolysis/tandem mass spectrometry

AUTHOR(S): Majumdar, Tapan K.; Ranasinghe, Asoka; Lu, Ling; Cooks, R. Graham; Fife, Wilmer K.; Zeldin, Martel

CORPORATE SOURCE: Dep. Chem., Purdue Univ., West Lafayette, IN, 47906, USA

SOURCE: Talanta (1993), 40(3), 363-79

CODEN: TLNTA2; ISSN: 0039-9140

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Desorption chemical ionization (DCI) and desorption electron ionization (DEI) of homo- and co-polymers of N-alkyl-4-vinylpyridinium triflates having Et, n-hexyl, and n-dodecyl groups as N-alkyl substituents, produce mass spectra that display oligomeric ions. These pos. charged ions are singly-charged and result from cleavage of the polymer into neutral oligomers and the loss of a single triflate anion per oligomer. Analogous neg. charged ions, in which each neutral oligomer carries an extra triflate anion, are observed in the desorption chemical ionization mass spectra. Each oligomer within the available mass range is represented in the mass spectra. The formation of cluster ions in which a single, multiply-charged cation is associated with a number of singly-charged anions, as observed for these ammonium polysalts, is unusual. Five major and three minor series of pos. charged ions are observed in DCI and DEI methods of ionization. Ions in the different series correspond either to cleavage at different bonds between the constituent monomers or to hydrogen transfer in different directions. Unique and structurally diagnostic fragmentation processes are observed in tandem mass spectrometry expts. performed using collision activated dissociation of mass-selected oligomeric ions.

L2 ANSWER 34 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1992:584189 CAPLUS

DOCUMENT NUMBER: 117:184189

TITLE: Pyrolysis-gas chromatography-mass spectrometry of a series of bile acid sequestrants

AUTHOR(S): Haskins, Neville J.; Eckers, Christine; Mitchell, Robert

CORPORATE SOURCE: SmithKline Beecham Pharm., The Frythe Welwyn Hertfordshire, AL6 9AR, UK

SOURCE: Analyst (Cambridge, United Kingdom) (1992), 117(9), 1413-16

CODEN: ANALAO; ISSN: 0003-2654

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Pyrolysis of a series of polymers based on polystyrene and used as bile acid sequestrants produced characteristic mixts. of compds. which were analyzed by gas chromatog.-mass spectrometry. The nature of the substituent groups was clearly apparent while the polymer backbone gave rise to representative styrenes. The reproducibility of the results was examined by experimenting with the temperature of pyrolysis. It was found that at low temps., very little fragmentation of the polystyrene backbone occurred,

but the substituents were still released in high yield. The orientation of the various substituted styrenes generated by pyrolysis was confirmed by the use of gas chromatog. with IR and mass spectrometric detection.

L2 ANSWER 35 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1991:559912 CAPLUS

DOCUMENT NUMBER: 115:159912

TITLE: "Mass spectral determination of cyclic oligomer distributions in polymerization and degradation reactions"

AUTHOR(S): *Montaudo, Giorgio*

CORPORATE SOURCE: Dip. Sci. Chim., Univ. Catania, Catania, 95125, Italy

SOURCE: **Macromolecules (1991), 24(21), 5829-33**

CODEN: MAMOBX; ISSN: 0024-9297

DOCUMENT TYPE: Journal

LANGUAGE: English

AB A discussion of the use of mass spectral data to determine the distributions of cyclic oligomers formed during polymerization and polymer degradation reactions is presented. The exptl. distributions of cyclic oligomers allow discrimination between thermodynamically and kinetically controlled cyclization processes. In the former case the cyclic oligomer concentration decreases with  $n-2.5$ , and in the latter with  $n-1.5$ . The exponent in the cycle distribution law indicates whether the cyclization process is occurring through a back-biting or an end-biting reaction.

L2 ANSWER 36 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1991:430209 CAPLUS

DOCUMENT NUMBER: 115:30209

TITLE: Microstructure of bacterial poly(<SYM98>-hydroxybutyrate-co-<SYM98>-hydroxyvalerate) by fast atom bombardment mass spectrometry analysis of their partial degradation products

AUTHOR(S): Ballistreri, A.; Garozzo, D.; Giuffrida, M.; Montaudo, G.

CORPORATE SOURCE: Inst. Chem. Technol. Polym. Mater., Univ. Catania, Catania, 95125, Italy

SOURCE: NATO ASI Series, Series E: Applied Sciences (1990), 186(Novel Biodegrad. Microb. Polym.), 49-64

CODEN: NAESDI; ISSN: 0168-132X

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The partial methanolysis and the partial pyrolysis of bacterially synthesized <SYM98>-hydroxybutyric acid-<SYM98>-hydroxyvaleric acid copolymers were performed and the oligomers produced were analyzed by fast-atom-bombardment mass spectrometry. An algorithm was developed to distinguish pure random copolymers from mixts. of random copolymers, using the relative abundances of the oligomers which could be deduced from the mass spectra.

L2 ANSWER 37 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1990:199294 CAPLUS

DOCUMENT NUMBER: 112:199294

TITLE: Thermal degradation and mass-spectrometric fragmentation processes of polyesters studied by time/temperature-resolved pyrolysis-field ionization mass spectrometry

AUTHOR(S): Plage, Bernd; Schulten, Hans Rolf

CORPORATE SOURCE: Dep. Trace Anal., Fachhochsch. Fresenius, Wiesbaden, D-6200, Germany

SOURCE: Macromolecules (1990), 23(10), 2642-8

CODEN: MAMOBX; ISSN: 0024-9297

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Linear polyesters of the lactone and the diol-dicarboxylic acid types were pyrolyzed in the ion source of a mass spectrometer. The gaseous degradation products were softly ionized by means of field ionization. Oligomers were the dominant products observed as protonated species. Distinct mass-spectrometric fragmentation occurred, forming singly charged and doubly charged carboxonium and hydroxonium ions, resp. Single fragments were formed with high intensity when the simultaneous formation of several double bonds is possible with the structural subunits present. In addition, cyclic 6-membered transition states and the possibility of the formation of stable cyclic fragments increased the degree of fragmentation.

L2 ANSWER 38 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1990:62439 CAPLUS

DOCUMENT NUMBER: 112:62439

TITLE: "Use of liquid chromatography and mass spectroscopy to select an oligomer representative of polyester hydrolysis pathways"

AUTHOR(S): Maniar, Manoj L.; Kalonia, Devendra S.; Simonelli, Anthony P.

CORPORATE SOURCE: Sch. Pharm., Univ. Connecticut, Storrs, CT, 06269, USA

SOURCE: Journal of Pharmaceutical Sciences (1989), 78(10), 858-62

CODEN: JPMSAE; ISSN: 0022-3549

DOCUMENT TYPE: Journal

LANGUAGE: English

AB An anal. method was developed for the simultaneous determination of a series of low-mol.-weight biodegradable polyesters and their degradation products. The separation is accomplished using a strong anion exchange HPLC column. Polyesters and their degradation products are identified by fast atom bombardment mass spectrometry. This technique will enable one to establish the polyester hydrolysis pathways and determine accurate kinetic parameters.

L2 ANSWER 39 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1989:534778 CAPLUS

DOCUMENT NUMBER: 111:134778

TITLE: Pyrolysis-GC and MS applied to study oligomer formation in the degradation of polystyrene and styrene copolymers

AUTHOR(S): Dean, Louise; Groves, Sally; Hancox, Robert; Lamb, Gordon; Lehrle, Roy S.

CORPORATE SOURCE: Dep. Chem., Univ. Birmingham, Birmingham, B15 2TT, UK  
SOURCE: Polymer Degradation and Stability (1989), 25(2-4), 143-60  
CODEN: PDSTDW; ISSN: 0141-3910

DOCUMENT TYPE: Journal; General Review

LANGUAGE: English

AB A review with 12 refs. on the use of pyrolysis-gas chromatog. and mass spectrometry to determine the mechanism of pyrolysis of polystyrene and styrene copolymers.

L2 ANSWER 40 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1989:440057 CAPLUS

DOCUMENT NUMBER: 111:40057

TITLE: Pyrolysis-field ionization mass spectrometry of aliphatic polyesters and their thermal interactions in mixtures

AUTHOR(S): Plage, Bernd; Schulten, Hans Rolf

CORPORATE SOURCE: Dep. Trace Anal., Fachhochsch. Fresenius, Wiesbaden, 6200, Fed. Rep. Ger.

SOURCE: Journal of Analytical and Applied Pyrolysis (1989), Volume Date 1988, 15, 197-207

CODEN: JAAPDD; ISSN: 0165-2370

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The thermal degradation of poly(ethylene succinate) (I), poly(butylene adipate) (II), poly(neopentyl succinate) (III), and mixts. of I with II or III was studied by the title method. Cyclic oligomers were observed as main products, accompanied by linear species terminated by olefinic, hydroxyl, or carboxonium end-groups. Carboxonium end-groups are only formed by mass spectrometric fragmentation. Abundant series of doubly charged ions were found. With the polymer mixts., at temps. below 250° the mass spectra contain the same signals as with the single components. In contrast, in the temperature range of the polymer backbone degradation (above 300°) mixed oligomers with random distribution of the polymer subunits are observed. This random distribution is also seen in the highly abundant doubly charged fragments and shows that in the upper temperature range thermal degradation occurs via intermol. ester exchange reactions.

L2 ANSWER 41 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1988:550174 CAPLUS

DOCUMENT NUMBER: 109:150174

TITLE: Thermal degradation of aliphatic polyamides studied by field ionization and field desorption mass spectrometry

AUTHOR(S): Schulten, Hans Rolf; Plage, Bernd

CORPORATE SOURCE: Abt. Spurenanal., Fachhochsch. Fresenius, Wiesbaden, D-6200, Fed. Rep. Ger.

SOURCE: Journal of Polymer Science, Part A: Polymer Chemistry (1988), 26(9), 2381-94

CODEN: JPACEC; ISSN: 0887-624X

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The aliphatic polyamides nylon 66 (I), 69, 610, 612, 126 (II), 1210, and 1212 of the diamine dicarboxylic acid-type were pyrolyzed in the ion source of a double-focusing mass spectrometer and the thermal degradation products were recorded by field ionization (FI) and field desorption (FD) mass spectrometry (MS). In the FI mode, several series of thermal degradation products differing in the number of polymer repeating units were detected <1000 Daltons. The main products were oligomers and, in addition, protonated dinitriles and various protonated nitriles were formed in large amts. except for I and II. These 2 polymers form, in contrast to all other samples, large amts. of protonated amines and diamines. The technique employed allowed distinction between oligomers already present in the original polymer and oligomers formed by thermal fission of bonds in the polymer chain. Reaction mechanisms were given that explained the products observed. High resolution expts. and accurate mass measurements were performed to confirm the proposed structures. In the FD mode, cationized oligomers (attached mostly to a sodium cation) were observed <200° with the dimers being the base peak for most samples. In contrast to the FI results, the monomers were only detected at very low intensities. Similarly, only weak signals for addnl. thermal degradation products were registered except for II. At higher temps. the FD mass spectra gave protonated and doubly protonated oligomers in the high mass range <SYM163>2000 Daltons, which resulted in complementary structural information about the polymers.

L2 ANSWER 42 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1988:529963 CAPLUS

DOCUMENT NUMBER: 109:129963

TITLE: Characterization of polysiloxanes by high-resolution pyrolysis-gas chromatography-mass spectrometry

AUTHOR(S): Fujimoto, Shinji; Ohtani, Hajime; Tsuge, Shin

CORPORATE SOURCE: Fac. Eng., Nagoya Univ., Nagoya, 464, Japan

SOURCE: Fresenius' Zeitschrift fuer Analytische Chemie (1988), 331(3-4), 342-50

CODEN: ZACFAU; ISSN: 0016-1152

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Thermal degradation of various polysiloxanes was studied by pyrolysis-gas chromatog. (GC) with a fused-silica capillary column. The peak identification was carried out by a directly coupled GC-mass spectrometry system. Generally, the siloxanes were thermally degraded to cyclic oligomers along with a small amount of the compds. formed through elimination and/or recombination of the pendant substituents. Most of the degradation products including possible stereo- and structural isomers were completely separated in the high-resolution pyrograms which reflected the original stereoregularities and sequence distributions in the polysiloxane chains. Relative peak intensities of the characteristic cyclic oligomers observed were interpreted in terms of the compns. of several siloxane copolymers.

L2 ANSWER 43 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1988:529803 CAPLUS

DOCUMENT NUMBER: 109:129803

TITLE: Primary thermal fragmentation processes in poly(ethylene oxalate) investigated by mass spectrometry

AUTHOR(S): Ballistreri, Alberto; Garozzo, Domenico; Giuffrida, Mario; Impallomeni, Giuseppe; Montaudo, Giorgio

CORPORATE SOURCE: Dip. Sci. Chim., Univ. Catania, Catania, 95125, Italy

SOURCE: Polymer Degradation and Stability (1988), 21(4), 311-21

CODEN: PDSTDW; ISSN: 0141-3910

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The primary thermal decomposition mechanism of poly(ethylene oxalate) (I) was investigated by pyrolysis-mass spectrometry. Several mass spectrometric techniques were used to identify compds. present in the pyrolysis mixture: comparison of electron impact and chemical ionization spectra, high-resolution accurate mass measurements, and tandem mass spectrometry (daughter- and parent-ion spectra). The results obtained indicate that intramol. exchange reactions predominate in the primary thermal fragmentation processes yielding cyclic oligomers up to tetramer. I, prepared by condensation polymerization, was shown by <sup>1</sup>H-NMR to contain 7% of diethylene glycol (II) units along the polymer chain. A small amount of cyclic oligomers containing II units was detected among the pyrolysis products from I.

L2 ANSWER 44 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1988:407268 CAPLUS

DOCUMENT NUMBER: 109:7268

TITLE: Comparison of styrene oligomers generated by polystyrene thermolysis and styrene oligomerization

AUTHOR(S): Alajbeg, Andja; Svob, Vladimir; Krvavac, Ante

CORPORATE SOURCE: INA - Res. Dev., Zagreb, Yugoslavia

SOURCE: Polimeri (Zagreb, Croatia) (1988), 9(1-2), 7-11

CODEN: PLMRDI; ISSN: 0351-1871

DOCUMENT TYPE: Journal

LANGUAGE: Serbo-Croatian

AB Combined gas chromatog.-mass spectrometry was used to study the composition and structure of styrene diads and triads generated by thermolysis of polystyrene in N at 500°, by spontaneous oligomerization of styrene monomer, and by oligomers from condensates of polystyrene (I) production. A series of 12 diads with mol. weight 180-220 and 1 triad were identified in the thermolysis products of I, with 2,4-diphenyl-1-butene dimer (II) and 2,4,6-triphenyl-1-hexene trimer (III) as predominant components. A series of dimers (mol. weight 208) among which compds. of alicyclic structure and a group of trimers (mol. weight 312) were predominant were generated during the spontaneous oligomerization of styrene. Only small amts. of II and III were present in the oligomerization products.

L2 ANSWER 45 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1986:553651 CAPLUS

DOCUMENT NUMBER: 105:153651

TITLE: Primary thermal fragmentation processes in poly(lactic acid) investigated by positive and negative chemical ionization mass spectrometry

AUTHOR(S): Garozzo, Domenico; Montaudo, Giorgio; Giuffrida, Mario

CORPORATE SOURCE: Dip. Sci. Chim., Univ. Catania, Catania, 95125, Italy

SOURCE: Polymer Degradation and Stability (1986), 15(2), 143-9

CODEN: PDSTDW; ISSN: 0141-3910

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The mechanism of thermal decomposition of poly(lactic acid) [104585-16-2] was studied by direct pyrolysis-mass spectrometry using electron impact (EI), pos. chemical ionization (CI) and neg. chemical ionization (NCI) methods. The thermally formed cyclic oligomers of lactic acid are not stable under EI conditions. The results obtained by CI and NCI indicate that intramol. exchange reactions predominate in the primary thermal fragmentation processes.

L2 ANSWER 46 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1986:443760 CAPLUS

DOCUMENT NUMBER: 105:43760

TITLE: Mixtures of cyclic oligomers of poly(lactic acid) analyzed by negative chemical ionization and thermospray mass spectrometry

AUTHOR(S): Garozzo, Domenico; Giuffrida, Mario; Montaudo, Giorgio

CORPORATE SOURCE: Dip. Sci. Chim., Univ. Catania, Catania, I-95125, Italy

SOURCE: Polymer Bulletin (Berlin, Germany) (1986), 15(4), 353-8

CODEN: POBUDR; ISSN: 0170-0839

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The distribution of the cyclic oligomers of poly(lactic acid) (I) formed both by pyrolysis of I and by equilibration with a catalyst was studied by the title method. The relative amts. of the oligomers present in the mixture were also determined by these methods.

L2 ANSWER 47 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1985:454765 CAPLUS

DOCUMENT NUMBER: 103:54765

TITLE: Thermogravimetric analysis/mass spectroscopy of a phenolic resole resin

AUTHOR(S): Prime, R. Bruce

CORPORATE SOURCE: IBM, San Jose, CA, 95193, USA

SOURCE: Polymer Preprints (American Chemical Society, Division of Polymer Chemistry) (1985), 26(1), 15-16

CODEN: ACPPAY; ISSN: 0032-3934

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Coupling of a tandem triple quadrupole mass spectrometer to a thermogravimetric analyzer (all under unified computer control) increases the amount of chemical information obtained from either method alone and provides a means of delineating complex thermally activated processes such as the cure and decomposition of phenolic



resins. Weight loss in Methylon 75108 [9074-30-0] phenolic resin (a mixture of mono-, di-, and trimethylol allyl Ph ethers and their oligomers and byproducts) occurs in 3 steps: the 1st associated with cure and the other 2 with decomposition. Structures of the pos. ions formed from evolved gases during cure at 100-300° indicated that the gases evolved can be explained largely by volatilization of starting materials and evolution of water and HCHO condensation products. Based on the loss of allyl groups observed, it was proposed that allyl functions as a thermally labile protecting group, and its loss plays a key role in cure. The majority of ions created from gases evolved at decomposition from 440-500° were tentatively identified as phenol and substituted phenols, suggesting that the predominant crosslinked structure is the condensation product of 2,4,6-trimethylolphenol which contains both methylene and methylene ether crosslinks. The weight loss at 520-600° was interpreted as pyrolysis of the char formed in the 1st decomposition

L2 ANSWER 48 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1985:221408 CAPLUS

DOCUMENT NUMBER: 102:221408

TITLE: Direct mass spectrometry of polymers. XII. Thermal fragmentation processes in poly(<SYM97>-amino acids)

AUTHOR: Ballistreri, Alberto; Giuffrida, Mario; Maravigna, Pietro; Montaudo, Giorgio  
CORPORATE Ist. Dip. Chim. Chim. Ind., Univ. Catania, Catania, 6-95125, Italy  
Journal of Polymer Science, Polymer Chemistry Edition (1985), 23(4), 1145-61

CODEN: JPLCAT; ISSN: 0449-296X

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The thermal fragmentation processes in poly(<SYM97>-amino acids) have been investigated by direct pyrolysis-mass spectrometry. The mass spectral data show that the pyrolytic breakdown of polyglycine [25718-94-9], polysarcosine [25951-24-0], and polyproline [25191-13-3] leads to the formation of cyclic oligomers. Polyalanine [25191-17-7], poly(phenylalanine) [25191-15-5], and polytyrosine [25619-78-7] decompose yielding compds. with olefin and nitrile end-groups. Finally, in the case of poly(<SYM103>-Me glutamic acid) [25086-16-2], the primary thermal process is the loss of MeOH with consequent formation, along the polymer chain, of pyroglutamic units, which yield cyclic dimer as main pyrolysis product.

L2 ANSWER 49 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1985:46392 CAPLUS

DOCUMENT NUMBER: 102:46392

TITLE: Pyrolysis-fluorescence spectroscopy, pyrolysis-mass spectrometry and pyrolysis-liquid chromatography of linear and crosslinked polystyrenes

AUTHOR(S): Lai, Shih Tse; Locke, David C.

CORPORATE Queens Coll., City Univ. New York, Flushing, NY, 11367, USA

SOURCE: Journal of Chromatography (1984), 314, 283-93

CODEN: JOCRAM; ISSN: 0021-9673

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Pyrolysis of linear polystyrene (I) [9003-53-6] and crosslinked divinylbenzene-ethylvinylbenzene copolymer [9043-77-0] was studied using mol. fluorescence spectroscopy, high-performance liquid chromatog. (HPLC) with UV-absorption and fluorescence-emission detectors, and mass spectrometry. The products showed mol. fluorescence and excimer emission. Styrene oligomers up to the dodecamer could be separated isocratically with isooctane eluent on a 10- $\mu$ m Al<sub>2</sub>O<sub>3</sub> column. Pyrolysis of linear I in an enclosed system was shown by HPLC to yield primarily monomers; pyrolysis in a continuous-flow system produced monomers but in addition oligomers up to at least the heptamer. Stepwise pyrolysis-mass spectrometry substantiated the HPLC results in more detail and also provided an indication of head-to-head microstructure in the predominantly head-to-tail I.

L2 ANSWER 50 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN  
ACCESSION NUMBER: 1984:531235 CAPLUS  
DOCUMENT NUMBER: 101:131235  
TITLE: Mass spectral characterization and thermal decomposition mechanism of alternating silarylene-siloxane polymers  
AUTHOR(S): Ballistreri, Alberto; Montaudo, Giorgio; Lenz, Robert W.  
CORPORATE Ist. Dip. Chim. Chim. Ind., Univ. Catania, Catania, 95125, Italy  
SOURCE: Macromolecules (1984), 17(9), 1848-54  
CODEN: MAMOBX; ISSN: 0024-9297  
DOCUMENT TYPE: Journal  
LANGUAGE: English

AB The thermal decomposition of two alternating silarylene-siloxane polymers was studied by direct pyrolysis into a mass spectrometer. Intramol. exchange reactions occurred during the primary thermal fragmentation processes causing the formation of cyclic compds. Only part of the thermal fragments had structures corresponding to the repeating unit of the polymers. The remaining pyrolytic products resulted from thermal rearrangement of the silarylene-siloxane polymer chain and had structures different from those of the original polymer repeating units. Both siloxane and p-phenylenedisilanol cyclic oligomers were found in the products.

L2 ANSWER 51 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN  
ACCESSION NUMBER: 1984:210590 CAPLUS  
DOCUMENT NUMBER: 100:210590  
TITLE: Direct mass spectrometry of polymers. Primary thermal fragmentation processes in aromatic polyesters  
AUTHOR(S): Foti, S.; Giuffrida, M.; Maravigna, P.; Montaudo, G.  
CORPORATE SOURCE: Ist. Dip. Chim. Chim. Ind., Univ. Catania, Catania, 95125, Italy  
SOURCE: Polymer Preprints (American Chemical Society, Division of Polymer Chemistry) (1984), 25(1), 88-9  
CODEN: ACPPAY; ISSN: 0032-3934  
DOCUMENT TYPE: Journal  
LANGUAGE: English

AB The thermal fragmentation of polyesters (m-COC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>O-m)<sub>n</sub>, (m-COC<sub>6</sub>H<sub>4</sub>O)<sub>n</sub>, (CO(CH<sub>2</sub>)<sub>x</sub>CO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>O-m)<sub>n</sub> (x = 2, 4), and (m-COC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>(CH<sub>2</sub>)<sub>y</sub>O)<sub>n</sub> (y = 2, 4, 6) was investigated by direct mass spectrometry. The primary fragmentation process was the formation of cyclic oligomers and the secondary process was the hydrolytic cleavage of the cyclic compds.

L2 ANSWER 52 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1984:121731 CAPLUS

DOCUMENT NUMBER: 100:121731

TITLE: Thermal degradation of poly[oxy-(2,6-dimethoxy-1,4-phenylene)]

AUTHOR(S): Jauhiainen, Tuure Pekka

CORPORATE SOURCE: Dep. Chem. Biosci., Univ. Joensuu, Joensuu, 80101, Finland

SOURCE: Thermochimica Acta (1984), 72(1-2), 257-62

CODEN: THACAS; ISSN: 0040-6031

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The thermal degradation of the title polymer [25667-13-4] was studied by TGA, IR spectrometry, and pyrolysis-gas chromatog.- mass spectrometry. Degradation initiated at chain ends by cleavage of 2,6-dimethoxy-2,5-cyclohexadiene-1,4-dione, 2,6-dimethoxyphenol, 3,5-dimethoxyphenol, and oligomers. Decomposition of the main chain occurred by statistical CO-bond breaking with simultaneous H transfer. Me groups migrated from OMe groups to benzene rings. MeO groups cleaved mainly as MeOH and methanal. The reliability of the different methods is discussed.

L2 ANSWER 53 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1984:7332 CAPLUS

DOCUMENT NUMBER: 100:7332

TITLE: Mass-spectrometric study of polyblock polysulfone-polydimethylsiloxane block copolymers

AUTHOR(S): Trokhova, S. Sh.; Nekrasov, Yu. S.; Sizoi, V. F.; Storozhuk, I. P.; Korshak, V. V.

CORPORATE SOURCE: Inst. Elementoorg. Soedin., Moscow, USSR

SOURCE: Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya (1983), (10), 2257-9

CODEN: IASKA6; ISSN: 0002-3353

DOCUMENT TYPE: Journal

LANGUAGE: Russian

AB Mass-spectrometric study of copolymers consisting of blocks of polysulfone, based on bisphenol A and diphenylene sulfone monomer or on the same plus propylene monomer, and di-Me siloxane revealed that their thermal degradation at 250-500° proceeds via cleavage of Si-O, Si-Me, and Si-H bonds, resulting in an array of linear and cyclic siloxane oligomers.

L2 ANSWER 54 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1982:616849 CAPLUS

DOCUMENT NUMBER: 97:216849

TITLE: Some aspects of the synthesis and characterization of special polymers and oligomers by olefin metathesis

AUTHOR(S): Stelzer, F.; Graiman, C.; Hummel, K.

CORPORATE SOURCE: Inst. Chem. Technol. Org. Stoffe, Tech. Univ. Graz, Graz, A-8010, Austria

SOURCE: Colloid and Polymer Science (1982), 260(9), 829-36

CODEN: CPMSB6; ISSN: 0303-402X

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Homopolymers of 2-norbornene and 2,3-bis(trifluoromethyl)-2,5-norbornadiene (I), copolymer of 2-norbornene and 1,5-cyclooctadiene, and copolymers of I and cyclopentene are prepared by ring-opening metathesis polymerization. The polymers are degraded by a cross-metathesis with trans-4-octene. Only I homopolymer [72920-56-0] is not degradable. The catalyst system is  $WCl_6/Me_4Sn$  for all reactions. The low-mol.-weight cyclic oligomers in the polymerization mixts. and the degrading products are identified by gas chromatog.-mass spectrometry. The degradation expts. reveal reactivity differences for the double bonds in the polymer backbone. The effects of the differences and preparation of polymers with special structures are discussed.

L2 ANSWER 55 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1982:493068 CAPLUS

DOCUMENT NUMBER: 97:93068

TITLE: Analysis of polymers by pyrolysis/chemical ionization mass spectrometry

AUTHOR(S): Conway, D. C.; Marak, Roman

CORPORATE SOURCE: Dep. Chem., Texas A and M Univ., College Station, TX, 77843, USA

Journal of Polymer Science, Polymer Chemistry Edition (1982), 20(7), 1765-74

CODEN: JPLCAT; ISSN: 0449-296X

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The addition polymers polystyrene [9003-53-6] and polypropylene [9003-07-0] and the condensation polymers poly(ethylene terephthalate) (I) [25038-59-9] and nylon66 (II) [32131-17-2] were pyrolyzed directly in the ion source of a mass spectrometer by chemical ionization to reduce fragmentation. The spectra of I and II were relatively easy to interpret because most ion peaks occurred at a mass corresponding to the ion formula  $M \pm H$  or  $M + 3H$ , where M is the formula of the fragment of the original polymer chain. Oligomer peaks were observed for the addition polymers. The intensities of the protonated dimer and monomer were measured as a function of time for styrene (III) and propylene (IV). Essentially all the protonated III dimer was produced by the reaction of protonated III monomer with III, but at least part of the protonated IV dimer was formed by proton transfer to IV dimer. Absence of the analogous reaction in III is presumably a result of the reduced volatility of III dimer.

L2 ANSWER 56 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1981:16234 CAPLUS

DOCUMENT NUMBER: 94:16234

TITLE: Analysis of styrene polymers by mass spectrometry with filament-heated evaporation

AUTHOR(S): Udseth, Harold R.; Friedman, Lewis

CORPORATE SOURCE: Dep. Chem., Brookhaven Natl. Lab., Upton, NY, 11973, USA

SOURCE: Analytical Chemistry (1981), 53(1), 29-33

CODEN: ANCHAM; ISSN: 0003-2700

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Mass spectrometry of polystyrene [9003-53-6] (nominal mol. weight 2100) is discussed. The polystyrene was evaporated from a probe filament heated at 1000°/s under both electron-impact (EI) and chemical-ionization (CI) conditions. Under EI conditions, extensive fragmentation and depolymn. were observed and fragments with d.p. <SYM163> 11 were detected. Under CI conditions, fragments with d.p. <SYM163> 27 were detected and spectra that approx. reproduced the oligomer distribution were obtained. Temperature measurements and activation energies of evaporation rates for many of the ionic species were also measured.

L2 ANSWER 57 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1980:408933 CAPLUS

DOCUMENT NUMBER: 93:8933

TITLE: Mass-spectrometric study of chemical transformations of filled polymer systems on the basis of phenolphthalein-phenol-formaldehyde oligomer and molybdenum disulfide under thermal and mechanical effects

AUTHOR(S): Korshak, V. V.; Gribova, I. A.; Krasnov, A. P.; Nekrasov, Yu. S.; Mazaeva, I. S.; Badaeva, M. M.; Sergeev, V. A.; Shitikov, V. K.

CORPORATE SOURCE: Inst. Elementoorg. Soedin., Moscow, USSR

SOURCE: Vysokomolekulyarnye Soedineniya, Seriya A (1980), 22(4), 739-44

CODEN: VYSAAF; ISSN: 0507-5475

DOCUMENT TYPE: Journal

LANGUAGE: Russian

AB Friction of MoS<sub>2</sub>-filled phenolphthalein-phenol-formaldehyde copolymer [26700-64-1], determined by mass spectrometry and gel-sol anal., gives significant changes in polymer fragments at <SYM163>200° and oxidative thermal degradation at the polymer-filler interface at >220°. Friction at 100° led to degradation of phenolphthalein and diphenylmethane units, and at 200° mainly to degradation of diphenylmethane units.

L2 ANSWER 58 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1980:59382 CAPLUS

DOCUMENT NUMBER: 92:59382

TITLE: The thermal degradation of polysiloxanes. II. Poly(methylphenylsiloxane)

AUTHOR(S): Grassie, N.; Macfarlane, I. G.; Francey, K. F.

CORPORATE SOURCE: Dep. Chem., Univ. Glasgow, Glasgow, G12 8QQ, UK

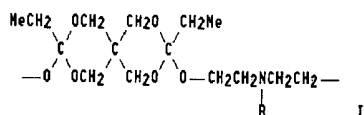
SOURCE: European Polymer Journal (1979), 15(5), 415-22

CODEN: EUPJAG; ISSN: 0014-3057

DOCUMENT TYPE: Journal

LANGUAGE: English

GI



AB Thermal anal. shows that poly(methylphenylsiloxane) is thermally more stable to weight loss than poly(dimethylsiloxane), the stability decreasing with increasing mol. weight and with replacement of terminal OH by Me<sub>3</sub>SiO groups. The main thermal degradation product is a mixture of all possible stereoisomeric cyclic trimers and tetramers together with small amts. of cyclic oligomer I [72140-38-6], cyclic oligomer II [72140-39-7], and pentamer. Benzene is also formed in a significant amount which increases with decreasing mol. weight and increasing temperature. The degradation mechanism is discussed in terms of dimethyl- and methylphenylsiloxane polymer structures, end groups, and chain branching.

L2 ANSWER 59 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1978:191734 CAPLUS

DOCUMENT NUMBER: 88:191734

TITLE: Thermal degradation of poly-<SYM101>-caprolactams (nylon 6)

AUTHOR(S): Luederwald, Ingo; Merz, Friedhelm; Rothe, Manfred

CORPORATE SOURCE: Inst. Org. Chem., Univ. Mainz, Mainz, Fed. Rep. Ger.

SOURCE: Angewandte Makromolekulare Chemie (1978), 67(1), 193-202

CODEN: ANMCBO; ISSN: 0003-3146

DOCUMENT TYPE: Journal

LANGUAGE: German

AB During pyrolysis of nylon 6 [25038-54-4] directly in the ion source of a mass spectrometer, cyclic oligomers present in the polymer begin to volatilize at <100° in the high vacuum and can be identified mass spectrometrically without previous isolation. At >390° under these conditions intense thermal degradation begins, giving mainly cyclic oligomers of caprolactam.

L2 ANSWER 60 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1977:423857 CAPLUS

DOCUMENT NUMBER: 87:23857

TITLE: Investigations of the structure of polyamides from cyclic dicarboxylic acids and piperazine by direct degradation in a mass spectrometer

AUTHOR(S): Foti, S.; Luederwald, I.; Montaudo, G.; Przybylski, M.

CORPORATE SOURCE: Org.-Chem. Inst., Univ. Mainz, Mainz, Fed. Rep. Ger.

SOURCE: Angewandte Makromolekulare Chemie (1977), 62(1), 215-27

CODEN: ANMCBO; ISSN: 0003-3146

DOCUMENT TYPE: Journal

LANGUAGE: German

AB Fragmentation reactions and thermal decomposition mechanisms were determined by pyrolysis of polyamides of 1,2-cyclobutane-, 1,2-cyclopentane-, and 1,2-cyclohexanedicarboxylic acids and piperazine in the ion source of a mass spectrometer.

Thermal degradation gave oligomers with piperazine endgroups and saturated and unsatd. cycloalkane endgroups.

L2 ANSWER 61 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1974:553027 CAPLUS

DOCUMENT NUMBER: 81:153027

TITLE: Investigations on polymers in the mass spectrometer. 4. Degradation reactions of polybenzyls

AUTHOR(S): Lenz, Robert W.; Luederwald, Ingo; Montaudo, Giorgio; Przybylski, Michael; Ringsdorf, Helmut

CORPORATE SOURCE: Inst. Org. Chem., Univ. Mainz, Mainz, Fed. Rep. Ger.

SOURCE: Makromolekulare Chemie (1974), 175(8), 2441-59

CODEN: MACEAK; ISSN: 0025-116X

DOCUMENT TYPE: Journal

LANGUAGE: German

AB Thermal degradation of poly(p-phenylene methylene) [27880-39-3] and poly[(2,5-dimethyl-p-phenylene)methylene] [28451-18-5] occurs by disproportionation and H transfer, giving monomeric and oligomeric products terminated by saturated or unsatd. (quinonemethide) groups. Degradation of poly(p-phenyleneethylidene) [32200-34-3] also gives vinyl-terminated products. Owing to steric hindrance, degradation of poly[(2,3,5,6-tetramethyl- p-phenylene)methylene] [28451-19-6] follows a different mechanism, and its thermal stability is lower.

L2 ANSWER 62 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1974:506206 CAPLUS

DOCUMENT NUMBER: 81:106206

TITLE: Field ion and electron impact mass spectrometry of polymers and copolymers. 3. Copolymers of <SYM97>-methylstyrene with methyl methacrylate and acrylonitrile

AUTHOR(S): Hummel, Dieter O.; Duessel, Heinz J.

CORPORATE SOURCE: Inst. Phys. Chem., Univ. Koeln, Cologne, Fed. Rep. Ger.

SOURCE: Makromolekulare Chemie (1974), 175(2), 655-65

CODEN: MACEAK; ISSN: 0025-116X

DOCUMENT TYPE: Journal

LANGUAGE: German

AB Pyrolytic field ion mass spectrometry demonstrated that methyl methacrylate-<SYM97>-methylstyrene polymer [25657-58-3] on pyrolysis decomp. primarily to the monomers. Unzippering depolymn. proceeds along the chain disregarding heterolinks between different monomers, with little formation of hetero- or homodimers. During pyrolysis of acrylonitrile-<SYM97>-methylstyrene polymer [25747-74-4], methylstyrene (I) sequences depolymerize by unzipping, forming I. Depolymn. stops at heterolinks, forming I-nitrile or I-(nitrile)<sub>2</sub> fragments. Long nitrile sequences, like polyacrylonitrile [25014-41-9], form nitrile oligomers and higher alkenenitriles. Isolated nitrile units form monomer.

L2 ANSWER 63 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1973:419357 CAPLUS

DOCUMENT NUMBER: 79:19357

TITLE: Mass spectrometer polymer studies. II. Degradation of poly-<SYM98>-alanines

AUTHOR(S): Leuderwald, I.; Ringsdorf, H.

CORPORATE SOURCE: Inst. Org. Chem., Univ. Mainz, Mainz, Fed. Rep. Ger.

SOURCE: Angewandte Makromolekulare Chemie (1973), 29-30, 453-70

CODEN: ANMCBO; ISSN: 0003-3146

DOCUMENT TYPE: Journal

LANGUAGE: German

AB The degradation reactions of poly-<SYM98>-alanines by direct pyrolysis in the mass spectrometer was investigated. In the pyrolytic step the amide nitrogen-<SYM98>-carbon linkage is cleaved preferentially, yielding oligomer <SYM98>-alanines with unsaturated and amide endgroups. These oligomers were ionized at the heteroatoms followed by <SYM97>N-, <SYM97>O-, <SYM98>O- and ketene fragmentation. The thermal degradation step and the fragmentation step in the ion source could be differentiated.

L2 ANSWER 64 OF 65 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1973:148551 CAPLUS

DOCUMENT NUMBER: 78:148551

TITLE: Identification of high polymers by thermal degradation in the mass spectrometer

AUTHOR(S): Zeman, A.

CORPORATE SOURCE: Unilever Forschungsges. m.b.H., Hamburg, Fed. Rep. Ger.

SOURCE: Therm. Anal., Proc. Int. Conf., 3rd (1972), Meeting Date 1971, Volume 3, 219-27. Editor(s): Wiedemann, Hans G. Birkhaeuser: Basel, Switz.

CODEN: 26LWAP

DOCUMENT TYPE: Conference

LANGUAGE: English

AB Ten com. polymers(e.g. polyethylene [9002-88-4], PVC [9002-86-2], rubber, a phenolic resin, and polystyrene [9003-53-6]) were degraded reproducibly in a high-vacuum mass spectrometer at 160-500.deg. to obtain characteristic mass spectra suitable for polymer identification. Additives and residual monomers in the polymer were fractionally evaporated and sep. analyzed before thermal degradation. Copolymers were differentiated from homopolymer mixts. by the presence of oligomeric fragment ions.

L4 ANSWER 1 OF 23 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 2004:480552 CAPLUS

DOCUMENT NUMBER: 141:76525

TITLE: *Simultaneous determination of drug surface concentration and polymer degradation kinetics in biodegradable polymer/drug membranes: a model drug delivery system*

AUTHOR(S): Lee, Joo-Woon; Gardella, Joseph A.

CORPORATE SOURCE: Department of Chemistry, The State University of New York at Buffalo, Buffalo, NY, 14260-3000, USA

SOURCE: Applied Surface Science (2004), 231-232, 442-446

CODEN: ASUSEE; ISSN: 0169-4332



PUBLISHER: Elsevier Science B.V.

DOCUMENT TYPE: Journal

LANGUAGE: English

AB This paper reports new simultaneous ToF-SIMS anal. to determine both the earliest stage of polymer degradation and the surface concentration of a drug additive. The static SIMS spectra of a model Ph<sub>3</sub>N/poly(L-lactic acid) (PLLA) (20:80 weight%) blend matrix (t .apprx. 0.4 <SYM109>m on 1.0 cm<sup>2</sup>) hydrolyzed in buffered conditions are simultaneously and independently analyzed in the low mass range for the surface accumulation profile of Ph<sub>3</sub>N and in the high mass for the hydrolytic degradation kinetics of PLLA, resp. The rate of PLLA degradation at pH 10.0 is approx. 2 times faster than that at pH 7.4, but the corresponding rate of Ph<sub>3</sub>N accumulation at the surface is accelerated by a factor of .apprx.10.5 times faster. The results provide new insight in evaluating the surface concentration of Ph<sub>3</sub>N (pK<sub>b</sub>=0) from the blends, indicating that the initial rapid increase in surface concentration of Ph<sub>3</sub>N is related to but not singularly dependent on the rate of PLLA degradation.

REFERENCE COUNT: 14

L4 ANSWER 4 OF 23 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 2003:505247 CAPLUS

DOCUMENT NUMBER: 139:197900

TITLE: "Simultaneous time-of-flight secondary ion MS quantitative analysis of drug surface concentration and polymer degradation kinetics in biodegradable poly(L-lactic acid) blends"

AUTHOR(S): Lee, Joo-Woon; Gardella, Joseph A., Jr.

CORPORATE SOURCE: Department of Chemistry, University at Buffalo, The State University of New York, Buffalo, NY, 14260-3000, USA

SOURCE: **Analytical Chemistry (2003), 75(13), 2950-2958**

CODEN: ANCHAM; ISSN: 0003-2700

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

LANGUAGE: English

AB This paper reports a new quant. method of analyzing both the earliest stage of degradation of a polymer and the surface concentration of an additive using time-of-flight secondary ion mass spectrometry ( TOF-SIMS). The static SIMS spectra of triphenylamine (Ph<sub>3</sub>N)/poly(L-lactic acid) (PLLA) (20:80 wt %) blend matrixes hydrolyzed in buffered conditions within a short-term (<48 h) period are simultaneously analyzed in the low-mass range for the surface accumulation profile of Ph<sub>3</sub>N and in the high-mass range to determine the hydrolytic degradation kinetics of PLLA, resp. The results provide new insight in evaluating the surface concentration of Ph<sub>3</sub>N (pK<sub>b</sub> <SYM187> 0) from the blends to see how it relates to the reactions (hydrolytic PLLA degradation) occurring in the surface region in the initial induction period over which negligible loss of polymer weight is observed The relative PLLA surface degradation at pH 10.0 is .apprx.2 times faster than that at pH 7.4. The relative extent of increase in Ph<sub>3</sub>N surface concentration assayed in pH 10.0 buffer system is 9 times greater than that at pH 7.4. The initial rapid increase in surface concentration of Ph<sub>3</sub>N is related to but

not singularly dependent on the rate of PLLA degradation at the surface of blend matrixes. REFERENCE COUNT: 30

L4 ANSWER 5 OF 23 CAPLUS COPYRIGHT 2004 ACS on STN  
ACCESSION NUMBER: 2003:307198 CAPLUS  
DOCUMENT NUMBER: 139:309632  
TITLE: Degradation of Disk Lubricant: Ramification in Disk Drives and Direct Detection by TOF-SIMS  
AUTHOR(S): Kasai, Paul H.; Raman, Vedantham  
CORPORATE SOURCE: IBM Research Division, Almaden Research Center, San Jose, CA, 95120, USA  
SOURCE: Tribology Letters (2003), 15(1), 15-28  
CODEN: TRLEFS; ISSN: 1023-8883  
PUBLISHER: Kluwer Academic/Plenum Publishers  
DOCUMENT TYPE: Journal  
LANGUAGE: English

AB The degradation of Z-dol catalyzed by Lewis acid centers on the slider surface leads to chain scission forming one type of fragment terminated with a fluorocarbonyl end-group and the other with a trifluoromethoxy end-group. The former, in contact with humid air, converts to a fluorinated carboxylic acid Z-COOH. Z-COOH is an excellent scavenger for alkali or alkaline earth metal ions. Z-COO-M<sup>+</sup> thus formed is a strong surfactant, and, in a humid environment, forms microdroplets embodying water in the core. Metal ions thus scavenged on a disk surface can be readily detected by TOF-SIMS, and the microdroplets by optical microscopy in the dark-field mode. The presence of fragments having a trifluoromethoxy end-group on the disk surface can also be established by TOM-SIMS. A careful intensity anal. of peaks due to anions having a trifluoromethoxy end-group permits a semi-quant. assessment of the extent of degradation. Also degradation is caused by such production processes as tape-polishing and by such disk drive operations as the head flying over a single track or over a band in a seek-mode. REFERENCE COUNT: 10

L4 ANSWER 6 OF 23 CAPLUS COPYRIGHT 2004 ACS on STN  
ACCESSION NUMBER: 2003:241159 CAPLUS  
DOCUMENT NUMBER: 139:199807  
TITLE: Analytical service of fuel cell  
AUTHOR(S): Fujiwara, Yutaka; Satake, Hajime; Ohashi, Kazutoshi  
CORPORATE SOURCE: Chiba Lab., Sumitomo Chemical Co., Ltd., Sodegaura, Chiba, 299-0266, Japan  
SOURCE: Denshi Zairyo (2003), 42(2), 81-84  
CODEN: DEZADL; ISSN: 0387-0774  
PUBLISHER: Kogyo Chosakai  
DOCUMENT TYPE: Journal; General Review  
LANGUAGE: Japanese

AB A review of anal. methods for evaluating degradation of polymer electrolytes in polymer electrolyte fuel cells, including FTIR, XPS, TOF-SIMS, TGA, and TG-MA.

L4 ANSWER 7 OF 23 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 2003:215865 CAPLUS

DOCUMENT NUMBER: 138:402363

TITLE: Analysis of polymer oxidation using  $^{18}\text{O}_2$  and TOF-SIMS

AUTHOR(S): Moeller, Kenneth; Jansson, Anna; Sjoval, Peter

CORPORATE SOURCE: Department of Chemistry and Materials Technology, SP Swedish National Testing and Research Institute, Boras, SE-501 15, Swed.

SOURCE: Polymer Degradation and Stability (2003), 80(2), 345-352

CODEN: PDSTDW; ISSN: 0141-3910

PUBLISHER: Elsevier Science Ltd.

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The authors have investigated oxidative degradation of low d. polyethylene (LDPE) by time-of-flight secondary ion mass spectrometry (TOF-SIMS). Replacing  $^{16}\text{O}_2$  in the exposure atmospheric by  $^{18}\text{O}_2$  makes it possible to identify species originating from the artificial aging, since O present in the material prior to aging can then be distinguished from O introduced in the aging process. The yield of O-ions in TOF-SIMS is very high, making oxygen-18 an excellent tool for following early stages of oxidative degradation. Furthermore, the authors have identified 2  $^{18}\text{O}$ -containing ions,  $\text{CH}_3^{18}\text{O}^+$  and  $\text{C}_2\text{H}_3^{18}\text{O}^+$ , which can also be used to follow the oxidation processes.

REFERENCE COUNT: 5

L4 ANSWER 8 OF 23 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 2003:97103 CAPLUS

DOCUMENT NUMBER: 139:296728

TITLE: "Analysis of the Initial Burst of Drug Release Coupled with Polymer Surface Degradation"

AUTHOR(S): Lee, Joo-Woon; Gardella, Joseph A., Jr.; Hicks, Wesley, Jr.; Hard, Robert; Bright, Frank V.

CORPORATE SOURCE: Department of Chemistry, University at Buffalo, The State University of New York, BuffaloNew York, NY, 14260-3000, USA

SOURCE: Pharmaceutical Research (2003), 20(2), 149-152

CODEN: PHREEB; ISSN: 0724-8741

PUBLISHER: Kluwer Academic/Plenum Publishers

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Local pH effect on the release of a model pH-inert hydrophobic drug coupled with polymer degradation is described at the induction phase of biodegradable polymer erosion for better understanding the nature of initial burst of a drug. Using a novel approach with time-of-flight secondary ion mass spectrometry, both surface concentration of  $\text{Ph}_3\text{N}$  and degradation kinetics of PLLA are simultaneously and independently determined from a model  $\text{Ph}_3\text{N}/\text{PLLA}$  (20:80%) blend matrix ( $t < 0.4 \text{ m}$  on  $1.0 \text{ cm}^2$ ). In vitro hydrolysis of the model blend matrix is investigated for short-term periods ( $< 24 \text{ h}$ ) at physiol. pH and temperature and compared to basic pH. The rate of PLLA degradation is accelerated by a factor of  $\approx 3$  when using basic pH in vitro, but the rate of  $\text{Ph}_3\text{N}$  accumulation at the surface

is accelerated by a factor of apprx.6. A new quant. method has been developed to examine the earliest stages of polymer degradation and drug release. It was applied to a model system that could not be examined by traditional in vitro methods. For the model system studied the release of a low mol. weight hydrophobic drug at the induction phase of polymer erosion is related to but not singularly dependent on degradation kinetics.

REFERENCE COUNT: 16

L4 ANSWER 10 OF 23 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 2001:921893 CAPLUS

DOCUMENT NUMBER: 136:232897

TITLE: "Thermal degradation of poly(ethylene oxide-propylene oxide-ethylene oxide) triblock copolymer: comparative study by SEC/NMR, SEC/MALDI-TOF-MS and SPME/GC-MS"

AUTHOR(S): *Gallet, Guillaume; Carroccio, Sabrina; Rizzarelli, Paola; Karlsson, Sigbritt*

CORPORATE SOURCE: Department of Polymer Technology, Royal Institute of Technology (KTH), Stockholm, SE-100 44, Swed.

SOURCE: **Polymer (2001), Volume Date 2002, 43(4), 1081-1094**

CODEN: POLMAG; ISSN: 0032-3861

PUBLISHER: Elsevier Science Ltd.

DOCUMENT TYPE: Journal

LANGUAGE: English

AB By comparing size exclusion chromatog./matrix assisted laser desorption ionization (SEC/MALDI) and SEC/NMR spectra from virgin poly(ethylene oxide-propylene oxide-ethylene oxide) triblock copolymer, we were able to understand the bimodal distribution observed in Poloxamer 407. Propylene oxide, isomerized to allyl alc. during polymerization, eventually forms a Poly(ethylene oxide-propylene oxide) diblock copolymer when EO is added to the feed. The oxidative thermal degradation of Poloxamer 407 at 80°C in air was studied. We found by MALDI that degradation starts after 21 days in the PPO block of the copolymer. This result was confirmed by solid phase microextraction/gas chromatog.-mass spectrometry (SPME/GC-MS): the first volatile degradation product to appear is 1,2-propanediol 1-acetate 2-formate. The structure of this mol. suggests that a six-ring intramol. decomposition reaction of the PPO chain occurs at the very beginning of the polymer breakdown. Thus, the secondary hydroperoxide formed on the PPO chain plays a major role on the thermooxidn. of poloxamer materials.

REFERENCE COUNT: 17

L4 ANSWER 13 OF 23 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 2000:366740 CAPLUS

DOCUMENT NUMBER: 133:120783

TITLE: "Time-of-flight secondary ion mass spectrometry studies of hydrolytic degradation kinetics at the surface of poly(glycolic acid)"

AUTHOR(S): *Chen, Jiaxing; Lee, Joo-Woon; Hernandez de Gatica, Norma L.; Burkhardt, Cindy A.; Hercules, David M.; Gardella, Joseph A., Jr.*

CORPORATE SOURCE: Department of Chemistry, SUNY at Buffalo, Buffalo, NY, 14260, USA

SOURCE: **Macromolecules** (2000), **33**(13), 4726-4732

CODEN: MAMOBX; ISSN: 0024-9297

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

LANGUAGE: English

AB This paper presents a new approach for studying the hydrolytic degradation kinetics of biodegradable polymers using time-of-flight secondary ion mass spectrometry (ToF SIMS). In this study, in vitro hydrolytic degradation of poly(glycolic acid) (PGA) has been carried out at 37°C in aqueous saline buffer solns. of different pH values. The mol. weight distribution of hydrolysis products was obtained from the ToF SIMS spectra. The average mol. weight of the hydrolysis products calculated from the ToF SIMS spectra is a function of hydrolysis time. Hydrolysis kinetics equations for PGA based on solid/liquid heterogeneous reaction conditions are established. A good linear relationship was obtained using these equations. The reaction rates observed in this study generally agree with observations reported in the literature. Kinetics consts. for the hydrolytic degradation of PGA at different pH values have been obtained. This new approach demonstrates that ToF SIMS can be a powerful and fast technique in the study of degradation kinetics of biodegradable polymers. Using ToF SIMS, the hydrolysis kinetics of biodegradable polymers can be explored in the range of hours with in-vitro methods. REFERENCE COUNT: 31

L4 ANSWER 15 OF 23 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1999:622905 CAPLUS

DOCUMENT NUMBER: 132:16871

TITLE: Ion migration effects in the degradation of polymeric electroluminescent devices

AUTHOR(S): Sheats, James R.; Chang, Ying-Lan; Roitman, Daniel

CORPORATE SOURCE: Hewlett Packard Laboratories, Palo Alto, CA, 94304, USA

SOURCE: ACS Symposium Series (1999), 735(Semiconducting Polymers), 144-162

CODEN: ACSMC8; ISSN: 0097-6156

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

LANGUAGE: English

AB We have analyzed degradation in polymer electroluminescent devices using time-of-flight secondary ion mass spectrometry (TOF -SIMS). The devices consisted of polyfluorene-based polymers cast on indium tin oxide (ITO) coated glass substrates, with evaporated calcium cathodes. The as-cast polymer films were found to have a significant number of pinholes. Indium, concentrated in spots, was found on the polymer surface in increasing amts. with increasing elec. stress. Similarly, the amount of residual calcium on the polymer surface (left after washing) increased with stress; in this case the metal ions were more uniformly distributed (but still inhomogeneous). The anode was found to be phys. roughened. We propose that the shorts commonly observed during initial operation of devices are related to cathode metal filling in pinholes, and the shorts that develop as a function of stress are caused by migration of indium ions toward the cathode and their reduction to a metallic pathway. REFERENCE COUNT: 28

L4 ANSWER 17 OF 23 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1999:308035 CAPLUS

DOCUMENT NUMBER: 131:51932

TITLE: "TOF-SIMS analysis of chemical state changes in cresol-novolac photoresist surface caused by O<sub>2</sub> plasma downstream"

AUTHOR(S): *Saito, Reiko; Ichinohe, Yuji; Kudo, Masahiro*

CORPORATE SOURCE: Manufacturing Engineering Research Center, Toshiba Corporation, Yokohama, Kanagawa, 235-0017, Japan

SOURCE: **Applied Surface Science (1999), 142(1-4), 460-464**

CODEN: ASUSEE; ISSN: 0169-4332

PUBLISHER: Elsevier Science B.V.

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The chemical state changes on cresol-novolac photoresist surfaces caused by O<sub>2</sub> plasma exposure were investigated using time-of-flight secondary ion mass spectrometry (TOF-SIMS). TOF-SIMS spectra were measured at the surface of the photoresist samples which were exposed to the O<sub>2</sub> plasma downstream for various exposure times and the spectral intensity changes of characteristic secondary ion species with respect to O<sub>2</sub> plasma conditions were quant. evaluated. Oxidation and decomposition of the polymer were observed from anal. of the changes in the peaks originating from the chemical structure of the polymer, both in pos. and neg. mass spectra. It was clarified that oxidation and vaporization attain a balance within a thin altered layer. In addition, depletion of photosensitive additive at the surface was confirmed and the usefulness of TOF-SIMS for predicting the reaction occurring on photoresist surfaces was successfully demonstrated. REFERENCE COUNT: 11

L4 ANSWER 18 OF 23 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1999:251037 CAPLUS

DOCUMENT NUMBER: 131:45452

TITLE: "Evaluation of resin by XPS and TOF-SIMS"

AUTHOR(S): *Sasakawa, Kaoru; Kurusu, Chie; Nakayama, Toshiro*

CORPORATE SOURCE: Kobelco Research Inst., Inc., Nishi-ku, Kobe, Hyogo, 651-2271, Japan

SOURCE: **Journal of Surface Analysis (1999), 5(2), 231-234**

CODEN: JSANFX; ISSN: 1341-1756

PUBLISHER: Hyomen Bunseki Kenkyukai

DOCUMENT TYPE: Journal

LANGUAGE: English

AB XPS and TOF-SIMS analyses have been performed for heated POM and ABS resins. In the case of POM, elimination of oxygen and formation of hydrocarbon species were observed after heating treatment. In the case of heated ABS, oxidation of polybutadiene and polystyrene occurred at the surface, and oxidation of only polybutadiene is observed at .apprx.0.1 mm depth from surface. Polyacrylonitrile showed no change by heating treatment.

REFERENCE COUNT: 6

L4 ANSWER 19 OF 23 CAPLUS COPYRIGHT 2004 ACS on STN  
ACCESSION NUMBER: 1997:345778 CAPLUS  
DOCUMENT NUMBER: 126:330999  
TITLE: Spectral analysis of eight polymers in SIMS by MO calculation. Prediction of cleavage of polymers and structural formula of the positive-ion fragment  
AUTHOR(S): Endo, Kazunaka; Hoshi, Takahiro; Kobayashi, Naoya; Miura, Hidetoshi; Kudo, Masahiro  
CORPORATE SOURCE: Tsukuba Research Laboratory, Mitsubishi Paper Mills, Ltd., Tsukuba, 300-42, Japan  
SOURCE: Polymer Journal (Tokyo) (1997), 29(5), 457-466  
CODEN: POLJB8; ISSN: 0032-3896  
PUBLISHER: Society of Polymer Science, Japan  
DOCUMENT TYPE: Journal  
LANGUAGE: English  
AB Spectra of eight polymers  $[(CH_2-CHR)_n$  ( $R = H, OH, F, Cl, OCH_3$ , and  $CONH_2$ ),  $(CF_2-CF_2)_n$ ] and  $(CH_2-CHCN)_n$  in time-of-flight (TOF)- and static(S)-secondary ion mass spectrometry (SIMS), resp., were analyzed by semiempirical and ab initio MO calcns. We predicted where the cleavage of the polymers occurs on sputtering, due to two-center bond energies of the model pentamers by semiempirical MO calcns. using AM1 program; a) the cleavage can occur in any bonds (polyethylene, poly(vinyl Me ether), polyacrylonitrile), b) the cleavage of the main chain occurs in any bonds, after side-chain groups cleaved first (poly(vinyl chloride), poly(acrylamide)), c) the main-chain carbons with the side-chain group cleave at any bonds of the main chain (poly(vinyl fluoride), poly(tetrafluoroethylene), poly(vinyl alc.)). We also determined the possible structural formulas of the secondary pos.-ion fragments in the range of 0-100 amu by ab initio MO calcns. using the HONDO7 program. REFERENCE COUNT: 12

L4 ANSWER 20 OF 23 CAPLUS COPYRIGHT 2004 ACS on STN  
ACCESSION NUMBER: 1996:57017 CAPLUS  
DOCUMENT NUMBER: 124:97679  
TITLE: "PMMA surface modification under keV and MeV ion bombardment in relation to mammalian cell adhesion"  
AUTHOR(S): Lhoest, J.-B.; Dewez, J.-L.; Bertrand, P.  
CORPORATE SOURCE: Centre de Res. en Materiaux Avances, Univ. Catholique de Louvain, Louvain-la-Neuve, 1348, Belg.  
SOURCE: Nuclear Instruments & Methods in Physics Research, Section B: Beam Interactions with Materials and Atoms (1995), 105(1-4), 322-7  
CODEN: NIMBEU; ISSN: 0168-583X  
PUBLISHER: Elsevier  
DOCUMENT TYPE: Journal  
LANGUAGE: English  
AB Spin casted poly(Me methacrylate) (PMMA) films were submitted to  $Ga^+$  (15 keV),  $Xe^+$  (4 keV) and  $He^+$  (1 MeV) ion bombardments with fluences varying between  $10^{+12}$  and  $10^{+16}$  ions/cm<sup>2</sup>. The surface modifications were studied by time-of-flight secondary ion mass spectrometry (ToF-SIMS) and XPS. The evolution of ToF-SIMS peaks characteristic of the polymer and of the degradation were followed as a function of the

ion fluence. Surface deoxygenation and loss of the methacrylate pendent group were observed. These results are discussed regarding the kind of energy deposition mode (electronic and nuclear). The XPS results confirmed the surface deoxygenation. Finally, the influence of the ion beam modifications on the adhesion of mammalian cells were investigated. For this purpose, the samples were reconditioned by a solution containing both a protein and a surfactant prior to inoculation with human epithelial cells in a serum free nutritive medium. The results showed a preferential cell adhesion in the bombarded areas.

L4 ANSWER 21 OF 23 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1995:4080 CAPLUS

DOCUMENT NUMBER: 122:134386

TITLE: "Probing the surface chemical structure of some novel poly(ortho esters) prepared with N-methyl- and N-phenylethanolamine by time-of-flight secondary ion mass spectrometry (ToF-SIMS)"

AUTHOR(S): *Davies, M. C.; Leadley, S. R.; Paul, A. J.; Vickerman, J. C.; Heller, J.; Franson, N. M.*

CORPORATE SOURCE: Dep. Pharm. Sci., Univ. Nottingham, Nottingham, NG7 2RD, UK

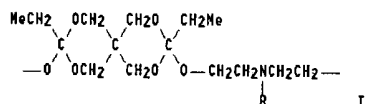
SOURCE: **Polymers for Advanced Technologies (1992), 3(6), 293-301**

CODEN: PADTE5; ISSN: 1042-7147

DOCUMENT TYPE: Journal

LANGUAGE: English

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AB The surface chemical structures of a series of novel poly(ortho ester) homopolymer and copolymer films (I) prepared using N-methyl- and N-phenyl-ethanolamine have been investigated using a time-of-flight secondary-ion mass spectrometry (ToF-SIMS). Systematic fragmentation patterns were observed within the ToF-SIMS spectra up to  $m/z = 1600$ , which were readily interpreted in terms of the polymer structures. Cations were detected which could be assigned to structures arising from the diols, the ortho ester species and diads, triads etc. of the monomer, from both the homopolymer and the random portion of the copolymer chains. The anal. of the proposed ion structures suggested two major mechanisms of fragmentation based on the cleavage of the ortho ester bonds involving either the exocyclic or the endocyclic alkoxy group. The ToF-SIMS peak area ratios for ions diagnostic of the diols and the ortho ester species reflected the bulk copolymer composition in a semi-quant. fashion. The observation of such relationships for high-mass cations (up to  $m/z = 500$ ) is particularly noteworthy and has not been reported previously. These studies allow a dramatic insight into the surface chemical structure of these poly(ortho esters)--information which has formed the foundation of current investigations on the mechanisms of the acid-catalyzed surface degradation of these polymers.

L4 ANSWER 22 OF 23 CAPLUS COPYRIGHT 2004 ACS on STN



ACCESSION NUMBER: 1994:631698 CAPLUS

DOCUMENT NUMBER: 121:231698

TITLE: "TOF SIMS in polymer surface studies"

AUTHOR(S): *Short, Robert D.; Ameen, A P.; Jackson, S T.; Pawson, D J.; O'Toole, L.; Ward, A J.*

CORPORATE SOURCE: Dep. Eng. Mater., Univ. Sheffield, Sheffield, S1 4DU, UK

SOURCE: **Vacuum (1993), 44(11-12), 1143-60**

CODEN: VACUAV; ISSN: 0042-207X

DOCUMENT TYPE: Journal; General Review

LANGUAGE: English

AB A review with 61 refs. describing advances made in the study of polymer surfaces at Sheffield University between 1988 and 1993. Over this period, we have taken advantage of the increased surface sensitivity, extended mass range, and imaging capability of Time-of-Flight Secondary Ion Mass Spectrometry (TOF SIMS) to investigate the surfaces of 'as-received', degraded, and modified polymers, as well as organic surface coatings prepared by plasma polymerization. Secondary ion mass spectrometry in polymer surface studies, surface structure of polymer blends, surface photo-oxidation of polystyrene (PS) and photostabilized PS, monitoring the effects of plasma surface treatment by TOF SIMS, and plasma polymer structure are reviewed and discussed. A brief overview of the developments in SIMS technol. and exptl. methodol. that now allow its routine use in polymer surface studies is also presented. The intention is not simply to draw attention to what we have already done, but to give an idea of where our studies are progressing. The work described is ongoing, indeed, in all the examples selected for this review, more potential areas of investigation have been opened than closed. Some weaknesses in the present use and understanding of SIMS, in polymer surface studies, are identified, and expts. designed to address these problems are described.

L4 ANSWER 23 OF 23 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1991:450693 CAPLUS

DOCUMENT NUMBER: 115:50693

TITLE: Time-of-flight static secondary ion mass spectrometry of additives on polymer surfaces

AUTHOR(S): Mawn, M. P.; Linton, R. W.; Bryan, S. R.; Hagenhoff, B.; Juergens, U.; Benninghoven, A.

CORPORATE SOURCE: Dep. Chem., Univ. North Carolina, Chapel Hill, NC, 27599-3290, USA

SOURCE: Journal of Vacuum Science & Technology, A: Vacuum, Surfaces, and Films (1991), 9(3, Pt. 2), 1307-11

CODEN: JVTAD6; ISSN: 0734-2101

DOCUMENT TYPE: Journal

LANGUAGE: English

AB A comparison of a polyethylene additive extract, deposited from solution as a monolayer on Ag, was made to an in-situ surface anal. of a bulk polyethylene sample using time-of-flight static secondary-ion mass spectrometry (TOF-SIMS). The in-situ anal. was able to confirm most of the additives detected in the extract. The additive mass range of 200-1200 was relatively free of spectral interferences from the polyethylene

matrix. High mass resolution studies were used to assist in the assignment of mol. identities of the additives. Deposition of standard additive solns. on Ag demonstrated the detection sensitivity of TOF-SIMS, illustrating the potential for mol. imaging of polymer domains as small as a few  $\mu\text{m}^2$ . The relative extent of oxidation of Naugard 524 antioxidant additive was also compared for a standard polyethylene extract and an in-situ anal. using the secondary-ion signals. The additive was more extensively oxidized after aging in the polymer matrix based upon measurement of the secondary-ion signal ratios.

	Hits	Search Text
1	2	("6,670,190").PN.
2	109	(degradation near2 kinetic\$3) with polymer\$3
3	2	((degradation near2 kinetic\$3) with polymer\$3) and "ToF SIMS"
4	2	("6670190").PN.
5	2	("6670190").PN.
6	1296	(436/173,34,85,).CCLS.